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A STUDY OF LC-39 CRYOGENIC SYSTEMS - FINAL REPORT

Paul R. Ludtke

Roland O. Voth



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VACUUM INSULATED TRANSFER LINES AT KENNEDY
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A STUDY OF LC-39 CRYOGENIC SYSTEMS-FINAL REPORT

**A Study of the Vacuum Insulated Transfer Lines
at Kennedy Space Center
(Part I)**

Paul R. Ludtke

**Cooldown Pressure Surges
(Part II)**

Roland O. Voth

**Cryogenics Division
Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302**

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**Technical Management
John F. Kennedy Space Center
Kennedy Space Center, Florida
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PREFACE

The work conducted under this contract is divided into two separate studies. Part I is an investigation of the cryo-pumping characteristics of the vacuum insulated transfer lines and the related vacuum measuring requirements at LC-39A and B. Part II is a study of the pressure surges incurred during cooldown of the liquid oxygen transfer lines.

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ABSTRACT

(Part I)

A study of the vacuum insulated liquid hydrogen and liquid oxygen transfer lines at Kennedy Space Center was conducted. A survey of other cryogenic facilities having large vacuum insulated transfer lines was made. The feasibility of using a condensing gas such as CO_2 inside the vacuum spaces to achieve a condensing-vacuum was evaluated. The type and amount of outgassing from the vacuum space components was determined, and the characteristics of palladium monoxide getter were investigated. The cryosorption characteristics of coconut charcoal and molecular sieve 5A were determined. The study indicates that at ambient temperature a maximum vacuum-space pressure of 4000 microns is acceptable for the LH_2 transfer lines, and a maximum vacuum-space pressure of 1000 microns is recommended for the LOX transfer lines. Various types of vacuum gauges were evaluated for use at KSC, and three types were recommended for consideration.

Key words: Activated charcoal; adsorbents; condensing-vacuum; cryosorption; getter; insulation; insulation-systems; molecular sieve; palladium monoxide; vacuum gauges; vacuum-jacketed transfer lines.

ABSTRACT

(Part II)

Future requirements for Launch Complex 39 at the Kennedy Space Center may change the cooldown procedure for the 14-inch cross-country liquid oxygen line. Because damaging pressure surges can develop due to the cooldown procedure, a simplified mathematical model was tested by comparing calculated pressure surges with measured surges for the current procedure. Although measured peak pressures were low, the mathematical model predicted both the magnitude and the time of the initial pressure surge. Because of the ability of the mathematical model to predict the peak cooldown pressure surge, proposed new procedures can be investigated using the model instead of using more expensive experimental tests.

Cooldown times for the uninsulated 14-inch cross-country line are also calculated, based on a "cold front" type model capable of including heat leak to the line during the cooldown period. Preliminary cooldown times are presented for various heat leak rates to the line and for two vent configurations.

Key words: Cooldown pressure surges; cooldown time; 14-inch lines; heat transfer; liquid oxygen; mathematical models; Saturn V ground support equipment; uninsulated line.

A STUDY OF THE VACUUM INSULATED TRANSFER LINES AT KENNEDY SPACE CENTER*

(Part I)

Paul R. Ludtke

1.0 Introduction

This is a final report under NASA Contract No. CC-59384 covering Phase II of the cryogenic transfer line study of the vacuum insulated liquid hydrogen and liquid oxygen transfer lines at Kennedy Space Center (KSC). Previous experience has shown that vacuum maintenance of the transfer line vacuum sections is costly. This program was initiated to determine what vacuum level could be tolerated before performing vacuum maintenance on the transfer line sections. Allowing the vacuum level to increase should decrease the vacuum maintenance costs. The performance of the vacuum probes and gauges presently being used at KSC are considered marginal. There are corrosion problems, and it is felt that the vacuum readings are not too reliable. A survey and evaluation of existing vacuum gauges for suitability in the present application were considered necessary.

1.1 Scope of the Study

The specific areas of study are:

- 1) Conduct a survey of other cryogenic facilities and determine their experience with large vacuum insulated transfer lines. Obtain pertinent information on allowable vacuum pressure levels, heat leak, condensing-vacuum applications, and vacuum gauge types and performance.

* This work was conducted at the National Bureau of Standards under the sponsorship of Kennedy Space Center-NASA.

- 2) Conduct a literature search to obtain and evaluate work on the effect of various gases at different pressure levels on the heat transfer through multi-layer insulation systems.
- 3) Evaluate the vacuum space pressure maintenance records for the various transfer line vacuum spaces at KSC. Prepare a summary and make recommendations concerning vacuum maintenance and pressure levels.
- 4) Study and evaluate the possibility of using a totally condensible gas such as CO_2 within the vacuum spaces of the existing transfer lines at KSC.
- 5) Determine the effect of increased vacuum space pressure on the total heat leak of the LOX and LH_2 transfer lines and recommend maximum tolerable vacuum space pressure levels for each system.
- 6) Study and evaluate the cryosorption characteristics of the sorbents within the vacuum spaces. Determine if the sorbents are capable of cryosorbing the gases under consideration from several-thousand microns pressure to an acceptable insulating vacuum (10^{-4} torr).
- 7) Survey and evaluate the various types of vacuum gauges available and suitable for use in the pressure range recommended, and in the corrosive environment at KSC.

Certain ground rules and assumptions have been made for this study and are defined as follows:

- 1) Most calculations were made using appropriate values for a typical 45-foot spool section of the LOX or LH_2 transfer lines.
- 2) Since the cross-country transfer lines are already fabricated and installed, the study to determine the effect of increased vacuum level was made assuming that very little or no modification would be made to the present vacuum jacketed transfer lines.

- 3) It was assumed that there is adequate multi-layer insulation within each vacuum space and that the heat transfer due to radiation and solid conduction is essentially constant, and cannot be improved without extensive modification to the system. Heat transfer by both of the above modes was considered fixed, and the heat transfer due to residual gas conduction was the only variable considered.
- 4) The task of determining if higher vacuum levels would be acceptable essentially boiled down to determining the cryosorption capability of the sorbents to cryosorb this increased quantity of gas. Considerable effort was expended in evaluating the cryosorption characteristics of the sorbents within the vacuum spaces, because these sorbents do the cryopumping which determines whether the residual gas conduction will be negligible or intolerable. The condition and capacity of these sorbents is most important.
- 5) It has been assumed that the vacuum spaces are vacuum pumped to a pressure level of 10-50 microns when they are serviced.
- 6) Certain pertinent information, concerning the details of vacuum space components, and methods and techniques of fabrication was unobtainable; it was assumed in these cases that the construction and fabrication details conformed to good cryogenic engineering practice.

1.2 Cryosorption Terminology

Some of the terminology in this report will be new to people who are not familiar with cryosorption terms. A brief explanation and some definitions are provided to help promote a better understanding of the report.

There are three basic mechanisms by which gas is taken up by a solid. These mechanisms are adsorption, absorption, and chemisorption. Adsorption is briefly defined as a process wherein gas is removed by physical attraction to the surface of a sorbent. Absorption is defined as the removal of gas by diffusion into the structure of the sorbent. Chemisorption is the removal of gas due to chemical combination with a solid by means of a chemical reaction. When speaking of cryosorption, it is not correct to refer to adsorption or absorption of a gas by a sorbent, because usually both of these mechanisms are involved when a gas is cryosorbed. Because of this, the term sorption will be used throughout this report to mean the removal of gas by both adsorption and/or absorption.

In addition, a distinction will be made between the terms getter and sorbent. A getter is often thought of as any substance that removes residual gas from an evacuated space. Substances such as activated charcoal, molecular sieves, and silica gel are often referred to as getters. However, in this report, the term getter will apply to a chemically active substance such as palladium monoxide (PdO), which removes gas from an evacuated space by chemical reaction only, and usually functions better at ambient or elevated temperatures. The term sorbent will be used to refer to substances such as activated charcoal, molecular sieves, silica gel, and other chemically inactive substances, which remove gas mainly by physical adsorption and usually exhibit greatly increased sorption capacities at cryogenic temperatures. A distinction should be made between these two mechanisms of removing residual gas from an evacuated space. One mechanism removes gas by an irreversible chemical reaction that increases with temperature, whereas the other removes gas by a reversible physical sorption process and the sorption rate increases at lower temperatures.

Most of the cryosorption terms used and a brief definition of each are given below.

Adsorption	Physical buildup or excess concentration of a gas on the surface of a solid due to Van der Waal's forces.
Absorption	The penetration of gas or vapor into the inner structure of a solid by a diffusional mechanism.
Chemisorption	Removal of gas due to chemical combination with a solid by means of combination, decomposition, or replacement type chemical reactions.
Sorption	General term which includes two mechanisms by which a gas is taken up by a solid--adsorption and absorption.
Sorb	To adsorb and/or absorb a gas.
Desorb	To remove sorbed gas from a sorbent, usually by vacuum pumping and/or heating.
Sorbent	A particulate material with very large surface area such as molecular sieve or coconut shell charcoal.
Cryosorb	To sorb gas on a sorbent at cryogenic temperatures.
Cryopump	To reduce the pressure inside a closed vessel or vacuum space by cryosorbing or condensing the gas.
Free Sorption	The sorption of atmospheric air and moisture onto an exposed sorbent.

Cryopumping Rate	The rate at which gas is condensed onto a cold surface or sorbed into a cold sorbent, or a combination thereof.
Condensing-Vacuum	The technique of using a gas, within a vacuum space, that will condense to solid on a cryogenic surface, with a very low vapor pressure.
Condensible Gas	A gas that will condense to the solid phase at the given operating temperature.
Non-Condensible Gas	A gas that will not condense to the solid phase at the given operating temperature.
Residual Gas	The gas at very low pressure (10^{-5} to 10^{-1} torr) within the vacuum space and the interstitial spaces of the multi-layer insulation.
Presorbed Gas	Gas which is sorbed by the sorbent prior to the primary cryosorption application.
Presorbed Water	Water which is sorbed by the sorbent, usually from the atmosphere at ambient temperatures, prior to the primary cryosorption application.
Ambient Temperature Vacuum-Pumpout Level	The ambient temperature pressure level within the vacuum space at which vacuum maintenance procedures dictate the vacuum space should be pumped.
Vacuum-Bakeout	A procedure wherein a vacuum space is dynamically pumped and simultaneously baked at a specified temperature for a given period of time.

Hydrogen-Bakeout	A vacuum-bakeout at 250-300°C for 15-20 hours to desorb most of the hydrogen gas from within the metal.
Palladium Monoxide	A compound in granular or powdered form which has the ability to convert hydrogen to water using a substitution type chemical reaction.
Specification Heat Leak	The heat leak given in the specifications for the manufacture of defined lengths of vacuum jacketed transfer line.
Equilibrium Sorption Capacity or Sorption Capacity	The amount of gas a given amount of sorbent will hold at equilibrium conditions--(partial pressure of the gas, sorbent temperature, and the type of gas).
Getter	A compound such as palladium monoxide that removes a certain type of gas from a vacuum space, usually by a chemical reaction. In contrast to a sorbent, a getter usually functions better at ambient temperatures.
Torr-Liter	One liter of gas at 1 torr pressure (or 10 liters of gas at 1/10 torr pressure, etc. . . .).
Micron-Liter	One liter of gas at 1 micron pressure (or 10 liters of gas at 1/10 micron pressure, etc. . . .).
Cm ³ [STP]	One cubic centimeter of gas at 760 torr and 273 K.
Insulating Vacuum	A pressure of 10 ⁻⁴ torr or less.

Micron	The pressure which will support a column of mercury 1 micron (10^{-6} m) high at 0°C.
Spool Section	A section of vacuum insulated transfer line 45 feet long.
Vacuum Section	The vacuum space of one spool section.
Outgassing Load	The amount of gas diffusing into a vacuum space from metal and other vacuum space components.
Pore Diameter (Molecular Sieves)	The diameter of the free apertures leading into the spherical cavities of the fundamental molecular sieve building block.
Pore Diameter (Coconut Charcoal)	The mean diameter of the long tubular passages extending into the body of the charcoal.

2.0 Survey of Vacuum Insulated Transfer Lines at Other Cryogenic Facilities

A number of other facilities involved with the transfer of cryogenic fluids were contacted in an effort to gather additional information concerning large vacuum insulated transfer lines. Questionnaires from and telephone conversations with people at these installations provided the needed information to gain a better understanding of problems associated with large vacuum insulated transfer lines. Specific questions asked were:

- 1) What is the ambient temperature vacuum-pumpout level?
- 2) What is the normal pumpout interval for good lines?
- 3) What type vacuum gauges are used and how reliable are they?
- 4) What specific problems have been encountered with the vacuum insulated lines?

- 5) Are any of your lines made of 36% nickel steel and have you encountered any trouble with welding 36% nickel steel?
- 6) Do you have any cryogenic transfer lines that employ a condensing gas for vacuum insulation?
- 7) What type, if any, multi-layer is used in the vacuum space?

The facilities contacted were:

- 1) Mississippi Test Facility (MTF)
- 2) Chrysler Corporation, Space Division
- 3) Wyle Laboratories
- 4) Los Alamos Scientific Laboratory
- 5) Aerojet-General Corporation
- 6) Amatek/Straza Industries
- 7) CVI Corporation
- 8) Cryogenic Engineering Company
- 9) Lockheed - Space Division.

2.1 Results of the Survey

The ambient temperature pressure level at which the vacuum spaces were pumped at the various installations ranged from 10 to 1000 microns pressure. The 1000-micron level was at the Mississippi Test Facility, and this seems to be a satisfactory pumping level for their large transfer line. None of the facilities which are similar in fabrication to those at KSC had a pumpout level above 1000 microns pressure.

Nothing conclusive can be said about the normal or average pumpout interval for a typical installation. The pumpout interval is highly dependent upon initial bakeout, type, amount, and condition of sorbent, the presence of getters, and the high vacuum workmanship exercised in fabrication. Some of the transfer lines considered in the

survey were built on site by available technicians and others were fabricated in the manufacturer's shop. Some lines had sorbents, multi-layer insulation, and palladium monoxide hydrogen getter, while others had nothing in the vacuum space. A better indication for a normal pumpout period may be determined from the vacuum maintenance study of the KSC transfer lines in section 4.

Most of the larger facilities used thermocouple type vacuum gauges. Thermocouple probes and gauges similar to the units at KSC are used at the Mississippi Test Facility. The probes are stainless steel and the personnel at MTF seem to be quite satisfied with their performance. They feel the probes are operating within the $\pm 2\%$ full scale specifications, which is adequate to indicate pressure trends. The environment at MTF is not as corrosive as at KSC and they probably have much less pin corrosion than at KSC.

The one specific problem encountered most was that of leaks developing at the annular seal between the inner and outer lines. This is evidently due to cyclic differential thermal contraction between the inner and outer line, placing excessive stress on the usually thin annular members. Materials selection, stress analysis, welding techniques, and material thickness are very important in the design of these seals.

One other problem area encountered at the Mississippi Test Facility was that of a stainless steel bellows in the inner line imploding. The bellows was fabricated of 3 or 4 plies. A small leak developed at the inner ply, liquid condensed between the plies, and upon warming, the pressure imploded the inner ply of the bellows creating a restriction in the transfer line.

Several people contacted had experienced trouble in welding 36% nickel steel and appeared to be very critical of 36% nickel steel for vacuum insulated transfer line applications. Considering the availability

of this steel, the increased price, and the welding problems encountered at various installations, it appears advisable to avoid the use of 36% nickel steel for vacuum insulated transfer line applications. However, recent advances in the state-of-the-art of welding 36% nickel steel has undoubtedly increased the reliability of the welds.

Not one person contacted knew of any cryogenic transfer line at their installation, or any other installation, currently employing a condensing gas such as CO_2 or N_2 in the vacuum space. Further discussion of this technique is given in section 3.

Various types of multi-layer insulation were used in the vacuum spaces of the transfer lines. The two most popular types of multi-layer appear to be aluminized Mylar[®] and aluminum foil with a glass cloth separator. Most of the newer transfer line installations do have multi-layer insulation.

2.2 Conclusions Derived from Survey

Other installations using vacuum insulated transfer lines are having problems similar to those at KSC--small leaks, annular seal failure, problems with 36% nickel steel welds. Similar installations have a pumpout level of 1000 microns or lower. Other installations are also using thermocouple gauges to measure pressure in the range of 10- 1000 microns pressure with satisfactory results, and there appears to be no application where a condensing gas such as CO_2 is used in the transfer line vacuum space.

[®] Dupont Trade Name. Precise specifications in this report of certain commercial products have been necessary to make the results of the work sufficiently meaningful. Identification of these products or the manufacturer by the Bureau of Standards in no way implies a recommendation or an endorsement by the Bureau.

3.0 Condensing-Vacuum Study

There is a technique of cryopumping, using a condensing gas such as CO_2 inside the vacuum space of a cryogenic transfer line, where upon cooling, the gas freezes to a solid with a vapor pressure far below the pressure of a good insulating vacuum. Carbon dioxide is one of the more desirable gases because it has a very low vapor pressure at cryogenic temperatures and it is nontoxic. The vapor pressure at 90 K is 4×10^{-6} torr and at 40 K it is 7×10^{-26} torr [Mullins, 1963]. This technique has the advantage of being able to maintain a vacuum space pressure just above atmospheric pressure at ambient temperature. Thus, in an application where there are long periods between flow operations, there is very little air leakage into the vacuum space and there is no vacuum maintenance of the vacuum space. The concept appears sound and maintenance free, but apparently is not being used in the industry. The reason the technique is not being used is that a good vacuum is still required with the CO_2 , and also it is difficult to place and maintain pure CO_2 in the vacuum space.

3.1 Experimental Work

An experimental program to determine the feasibility and characteristics of using condensing-vacuum insulation was conducted at the NBS Cryogenic Division by Van Gundy [1960]. Approximately 100 different gases were evaluated for this specific application and only three were deemed potentially suitable. These were carbon dioxide (CO_2), sulfur dioxide (SO_2), and nitric oxide (NO). Carbon dioxide was chosen for experimental evaluation because it is more readily available in rather pure form, and it is not a severely toxic gas like sulfur dioxide.

Results of the NBS experimental program showed that it is very important to provide a matrix at the cold inner line to mechanically hold the frozen CO_2 , which resembles loose, fragile snow. If no matrix is provided, these particles are easily dislodged by slight vibration, allowing them to fall onto the warm outer surface and sublime. One of the better matrix configurations was found to be a 1-inch layer of fiber glass insulation covered with perforated aluminum foil. The perforations improved the gas flow to the cold surface, and the aluminum foil decreased the radiant heat to the inner line.

Early in the program, it was found that the CO_2 had to be very pure and a purification apparatus was built especially for this purpose. It was also necessary to vacuum-bake the copper transfer lines to remove contamination, moisture, and reduce the hydrogen outgassing rate. Recent work by Young [1968] has shown that the hydrogen outgassing rate from stainless steel and aluminum can be reduced by two orders of magnitude by a vacuum-bakeout at $250 - 300^\circ \text{C}$ for 15 - 20 hours. The same should be true of copper. Experiments at NBS showed that an ambient temperature transfer line vacuum space pressurized to 6 psig with CO_2 would cryopump to 10^{-5} torr at 20 K. It was also determined that the solid CO_2 at 20 K has the capacity to sorb large quantities of hydrogen gas. Their experiments showed that the amount of hydrogen gas which can be tolerated in the vacuum space was increased by a factor of 40 to 200 by the addition of CO_2 . The amount of hydrogen sorbed by the CO_2 at 76 K was negligible. Hydrogen gas would be present in most transfer line vacuum spaces due to continual metal outgassing.

Tempelmeyer [1970] has done some recent work on cryosorbing hydrogen gas on solid CO_2 deposits in the temperature range 12 - 22 K. He was able to form solid CO_2 sorbents which could sorb one hydrogen molecule for every two predeposited CO_2 molecules, or about 2.3 weight percent. Thus, solid CO_2 does have the ability to sorb a fair amount of hydrogen gas at temperatures near and below 20 K.

As a result of this program, some 1-inch nitrogen transfer lines using CO_2 as a condensing-vacuum were fabricated at NBS and put into service at Marshall Space Flight Center, and the lines were reportedly good. However, soon after the above program was completed, a commercial firm contracted to build similar transfer lines, using the CO_2 condensing-vacuum concept according to NBS specifications, but those transfer lines would not meet the heat leak specifications. Litigation ensued because the transfer lines would not meet the required specifications.

During the survey of other facilities, a question specifically asked was, "Do you have any, or know of any, cryogenic transfer lines using the condensing-vacuum principle, or have you had any previous experience with this technique?"

Representatives of all major cryogenic transfer line manufacturers were contacted, in addition to other large facilities where transfer lines using this principle might be installed. Not one person questioned knew of any transfer lines currently in existence using the condensing-vacuum principle. However, two people had tried using this principle. Martindale [1970] of Amatek/Straza Industries indicates that their firm conducted a small in-house program to further investigate the possibility of using this technique for cryogenic transfer lines. They had very poor luck in obtaining an insulating vacuum. He commented that there are two main problems: 1) Getting pure CO_2 inside the

vacuum space, and 2) the hydrogen outgassing from the metal transfer lines. The program was abandoned and Martindale expressed little hope for a field type installation subject to leaks at reduced pressure (when cold) and field type fabrication techniques.

Wyle Labs [Wheelock, 1970] used CO_2 for a condensing-vacuum application at 76 K. They used an 8-foot long section of 3-inch inner line. The inner line was covered with two layers of fiberglass and then wrapped with a layer of perforated aluminum foil, similar to the NBS lines. The purity of the CO_2 was unknown and the vacuum lines may not have been baked. The resulting vacuum level was very poor, and they never tried this concept again.

3.2 Evaluation of Experimental Work

After reviewing the experimental work conducted at this laboratory and talking to other people having experience with this technique, the following conclusions and recommendations seem appropriate.

It appears that the concept of using CO_2 for a condensing-vacuum in a cryogenic transfer line vacuum space is sound and the principle will work. However, it also appears to be just as important to exercise good high vacuum techniques with a condensing-vacuum application, as with conventional high vacuum transfer lines.

The following procedures must be adhered to:

- 1) A mechanical matrix must be provided for the solid CO_2 to adhere to.
- 2) The transfer line should be vacuum-baked to significantly reduce the initial hydrogen outgassing and decrease the long-term hydrogen outgassing. This is evidently quite important if the transfer line is to operate near or above 76 K.

- 3) Very pure CO_2 must be used.
- 4) Once the CO_2 is purified, it must then be transferred into the vacuum space without further contamination.
- 5) The vacuum space must be leak free. Any leak in the system would allow non-condensibles to enter when the line is cold and spoil the insulating vacuum.

Considering the above recommendations, it is quite evident that obtaining a good insulating vacuum (10^{-4} or less) using the condensing-vacuum technique requires as much care, if not more, than a conventional first-class high vacuum transfer line using multi-layer insulation, sorbents, and getters, in which the state of the art is well advanced. Also, the transfer line must be specifically constructed for a CO_2 application. Moreover, the CO_2 is no answer to leakage problems. Leakage cannot be tolerated in either of the systems.

It is conceivable that vacuum maintenance of a condensing-vacuum complex could be more work and more costly than a conventional high vacuum complex. There would still be leakage problems plus the added task of maintaining pure CO_2 in all vacuum spaces. No facility has experienced this type of maintenance to date, so the extent of the maintenance required is not known.

3.3 KSC Transfer Lines

The transfer lines at KSC were not designed for a condensing-vacuum. The inner line has no matrix system to hold the solid CO_2 . There is multi-layer insulation which could conceivably serve this purpose, but there is no experimental verification to substantiate this, and most likely, the multi-layer would not work as well as a fiber-glass matrix.

It is doubtful that any of the transfer lines at KSC were given a good hydrogen bakeout after fabrication. Young [1968] has shown that a vacuum-bakeout for 15 hours at temperatures no less than 250°C is necessary to effectively reduce the H₂ outgassing rate to a level of 10⁻¹² torr liter/cm² - sec. All of the vacuum jacketed transfer lines at Complex 39B were baked for 3 to 4 days to remove moisture and fingerprint type contamination, but this was not a strict hydrogen bakeout. The lines containing aluminized Mylar multi-layer insulation were baked at 135°C and the LOX lines with aluminum foil and glass paper were baked at 150 - 200°C [Martindale, 1970].

The transfer lines at Complex 39A were evidently not baked at all, although no precise information is available as to whether they were or were not baked. In either case, it appears that hydrogen outgassing could be above that expected from a system given a good hydrogen vacuum-bakeout.

Some of the vacuum spaces at KSC have leakage problems; however, leakage would continue to be a problem if the lines were pressurized with CO₂. As previously explained, CO₂ is not an answer to small leakage problems.

The purity of the available CO₂ should also be considered. Coleman grade of 99.99% purity is not too costly. This grade is available in the standard size 1A cylinder at a price of \$41.00 for 526 ft³ [STP]. This amounts to 8 cents/ft³ or 72 cents/spool section. This is the highest purity that can be purchased in the large cylinders. The next higher purity of 99.995% is not available in the large cylinders and is considerably more expensive. A typical analysis of the coleman grade gas would be similar to the analysis below for the 99.995% purity CO₂.

Total hydrocarbons	< 20 ppm
N ₂	< 40 ppm
O ₂	< 10 ppm
H ₂	< 10 ppm
CO	< 40 ppm
H ₂ O	< 1 ppm

The impurities most prevalent are N₂ and CO. Both gases are non-condensable at 90 K. Hydrogen is the only non-condensable at 20 K, but the quantity is small and would probably be sorbed on the solid CO₂ or the other sorbent in the vacuum space.

Thus, it appears that the coleman grade gas might require further purification for a condensing-vacuum application at 90 K. At 20 K there are very few non-condensibles and this purity gas would probably be sufficient.

Since the performance of coleman grade CO₂ appears marginal at 90 K, the effect of the CO₂ on the sorption capacity of the molecular sieves was considered. The presence of CO₂ near atmospheric pressure within the vacuum space could conceivably saturate most of this sorbent, since molecular sieves have a very strong attraction for CO₂.

The equilibrium sorption capacity of molecular sieve 5A for CO₂ at 800 torr and 25°C is 19 lbs of CO₂/100 lbs of molecular sieves [Union Carbide-b, 1970]. The sorption capacity increases as the temperature decreases. At the freezing point of CO₂ (195 K), the equilibrium sorption capacity is ≈ 30 wt percent (30 lbs of CO₂/100 lbs of molecular sieve sorbent). Assuming that equilibrium conditions exist, the added sorption capacity at 195 K has the effect of reducing the vacuum space pressure from 800 torr to ≈ 530 torr.

The remainder of the CO_2 would probably be frozen, rather than sorbed by the molecular sieves. At 30 weight percent, it is very safe to assume that the molecular sieves are saturated, and there would be little additional sorption of N_2 or CO by the molecular sieves. From this data, we can conclude that if either a LOX or LH_2 vacuum space containing molecular sieves is pressurized with CO_2 and later cooled below 195 K, the sieves are essentially saturated and have very little capacity for additional sorption of other gases.

The use of CO_2 in the vacuum spaces of the transfer lines at KSC is not recommended for the following reasons: 1) other people have had little success with CO_2 in systems not specifically designed and baked for a condensing-vacuum application, 2) information pertaining to the use of CO_2 in large field type transfer line installations is not available, 3) the lines at KSC were not specifically designed for a condensing-vacuum, 4) getting pure CO_2 inside the vacuum space is not easy, 5) transfer line maintenance could be more of a problem than now exists, and 6) the state of the art in manufacturing first class high vacuum transfer lines is well developed and the major portion of the transfer line sections at KSC perform well. If sufficient interest still exists in spite of the uncertainties mentioned in this section, it would be advisable to initiate a small experimental program, using a transfer line section similar to those installed at KSC, to determine if an insulating vacuum of 10^{-4} torr or less can be obtained in the vacuum spaces pressurized with CO_2 : upon cooling the inner line, typical maintenance data could be acquired.

4.0 Vacuum Space Components

The type and amount of materials inside a vacuum space are very important in trying to evaluate the performance of a sealed vacuum system. The type and amount of sorbent, the type of multi-layer insulation, the presence of a getter material, the amount of plastic, whether or not the system was baked, and the vacuum space dimensions are all important parameters. The following information was given for each type of vacuum jacketed transfer line at launch Complexes 39A and 39B. Where exact information was not obtainable, a reasonable assumption was made.

- (1) Amount and type of multi-layer insulation
- (2) Amount and type of sorbent used
- (3) Amount, type, and location of hydrogen getter
- (4) Amount and type of plastic line spacers
- (5) Area of the inner line exposed to vacuum space
- (6) Area of the outer line exposed to vacuum space
- (7) Volume of annular space
- (8) Volume of multi-layer insulation
- (9) Volume of the vacuum space (annular space less insulation volume)
- (10) Ratio of vacuum space volume to sorbent mass (liters/gm)
- (11) Type of joint between 45-foot spool sections
- (12) Bakeout treatment given vacuum sections.

The volume of the sorbent and the transfer line spacer assemblies were neglected when calculating the vacuum space volumes. The thickness of all glass paper was assumed to be 0.004 inches, and the thickness of all aluminum foil was assumed or known to be 0.00025 inches. The location of the sorbent within the vacuum spaces is not known. Preferably, it is dispensed along the line at spaced intervals. It is most

important to have good thermal contact between the sorbent and the inner line and it will be assumed that good thermal contact does exist.

4.1 10-Inch Vacuum Jacketed LH_2 Transfer Lines (Complex 39B)

- (1) 20 layers of multi-layer insulation (.00025 in. thick Mylar, aluminized on one side) [Martindale, 1970].
- (2) 1058 grams of activated coconut charcoal [Martindale, 1970].
- (3) No hydrogen getter used in the main vacuum space [Martindale, 1970].
- (4) Exact type of spacers unknown; it is assumed they are very similar to 39A specifications, (25% glass-filled fluorocarbon plastic pins).
- (5) Area of inner line = $17,560 \text{ in.}^2 = 122 \text{ ft}^2$.
- (6) Area of outer line = $20,238 \text{ in.}^2 = 141 \text{ ft}^2$.
- (7) Volume of annular space = $15,600 \text{ in.}^3 = 9.03 \text{ ft}^3$.
- (8) Volume of multi-layer insulation = 90 in.^3 .
- (9) Volume of vacuum space = $15,510 \text{ in.}^3 = 8.98 \text{ ft}^3 = 254 \text{ liters}$.
- (10) Ratio of vacuum space volume to sorbent mass = $254/1058 = 0.24 \text{ l/g}$.
- (11) Joint between spool sections is a sealed vacuum space containing palladium monoxide and 20 layers of aluminized Mylar insulation. The covers are welded to the outer line.
- (12) Individual spool sections were baked 3 - 4 days at 135°C [Martindale, 1970].

4.2 10-Inch Vacuum Jacketed LH_2 Transfer Lines (Complex 39 A)

- (1) The multi-layer insulation is aluminum foil and glass paper. For calculations, it was assumed that the aluminum foil is 0.00025 in. thick, the paper is 0.004 in. thick, and there are 20 layers of each.

- (2) The sorbent is molecular sieve 5A and the amount is unknown. For calculations, the amount is assumed to be 1000 grams.
- (3) Palladium monoxide was placed in the main vacuum space. The amount is not known but considered to be adequate for 5 years.
- (4) Plastic spacers are six 25% glass-filled fluorocarbon plastic pins with an outgassing area of 37 cm^2 .
- (5) Area of the inner line = $17,560 \text{ in.}^2 = 122 \text{ ft}^2$.
- (6) Area of the outer line = $20,238 \text{ in.}^2 = 141 \text{ ft}^2$.
- (7) Volume of annular space = $15,600 \text{ in.}^3 = 9.03 \text{ ft}^3$.
- (8) Volume of multi-layer insulation = 1528 in.^3 .
- (9) Volume of vacuum space = $14,072 \text{ in.}^3 = 8.14 \text{ ft}^3 = 231 \text{ liters}$.
- (10) Vacuum space to sorbent ratio = $231/1000 = 0.231 \text{ l/g}$.
- (11) Dresser type coupling over outer line at spool joints, no vacuum insulation at joints.
- (12) Bakeout information not available--it is assumed no bakeout was given these sections.

4.3 6-Inch Vacuum Jacketed LOX Transfer Lines (Complex 39B)

- (1) The multi-layer insulation consists of 20 layers of 0.00025 in. thick aluminum foil with glass paper for spacer material. [Martindale, 1970]. Exact paper thickness is not known but assumed to be 0.004 in. for calculations.
- (2) The sorbent is molecular sieve 5A and there are 1600 grams/45 ft spool section [Martindale, 1970].
- (3) Palladium monoxide was placed inside the vacuum space. The exact amount is not known, but is assumed to be adequate for 5 years.

- (4) Exact type of spacers is unknown; it is assumed they are very similar to 39A specifications (25% glass-filled fluorocarbon plastic pins).
- (5) Area of inner line = $11,029 \text{ in.}^2 = 76.6 \text{ ft}^2$.
- (6) Area of outer line = $13,868 \text{ in.}^2 = 96.3 \text{ ft}^2$.
- (7) Volume of annular space = $10,584 \text{ in.}^3$.
- (8) Volume of multi-layer insulation = 972 in.^3 .
- (9) Volume of vacuum space = $9,612 \text{ in.}^3 = 5.56 \text{ ft}^3 = 158 \text{ liters}$.
- (10) Vacuum space volume to sorbent ratio = $158/1600 = 0.098 \text{ l/g}$.
- (11) The joint between spool sections is a sealed vacuum space containing palladium monoxide. The covers are welded to the outer line.
- (12) The individual spool sections were baked 3 - 4 days at $150 - 200^\circ\text{C}$ [Martindale, 1970].

4.4 6-Inch Vacuum Jacketed LOX Transfer Lines (Complex 39A)

- (1) The multi-layer insulation is aluminum foil and glass paper. The thickness and number of layers is not known. For calculations, it was assumed that the aluminum foil is 0.00025 in. thick, the paper is 0.004 in. thick, and there are 20 layers of each.
- (2) The sorbent is assumed to be molecular sieve 5A. The exact amount is unknown and it will be assumed the amount is the same as in Complex 39B, i. e., 1600 gms/45-foot spool section.
- (3) Palladium monoxide was placed inside the vacuum space. The exact amount is unknown, but assumed to be adequate for 5 years.
- (4) Six 25% glass-filled fluorocarbon plastic spacers.

- (5) Area of inner line = $11,029 \text{ in.}^2 = 76.6 \text{ ft}^2$.
- (6) Area of outer line = $13,868 \text{ in.}^2 = 96.3 \text{ ft}^2$.
- (7) Volume of annular space = $10,584 \text{ in.}^3$.
- (8) Volume of multi-layer insulation = 972 in.^3 .
- (9) Volume of vacuum space = $9.612 \text{ in.}^3 = 5.56 \text{ ft}^3 = 158 \text{ liters}$.
- (10) Vacuum space volume to sorbent ratio = $158/1000 = 0.098 \text{ l/g}$.
- (11) Dresser type coupling over outer line at spool joints, no vacuum insulation at joints.
- (12) Bakeout information not available. It is assumed no bakeout was given these sections.

Some of the information concerning the vacuum space components and bakeout treatment was not specified on the drawings or in the specifications and was essentially impossible to obtain. It would be more precise to know the exact amount of molecular sieves inside the vacuum spaces of the transfer line at Launch Complex 39A. However, it is felt that a reasonable assumption was made, and the cryosorption calculations reveal that the exact amount of sieves is not critical.

It will be shown later that the vacuum spaces operating at 20 K have an ample amount of sorbent and the vacuum spaces operating at 90 K would not be helped much by an increased amount of molecular sieve.

5.0 Transfer Line Evaluation

In this section, the outgassing characteristics of the various transfer line components will be discussed and evaluated. An attempt will be made to calculate the outgassing rate for a typical vacuum space at KSC, and the advantages of baking a vacuum system will be discussed. The advantage of using a hydrogen getter such as palladium monoxide (PdO) will be given and the characteristics of the compound will be discussed. The vacuum maintenance records for the liquid oxygen and hydrogen lines of LC-39 will be evaluated, and vacuum maintenance recommendations will be made.

5.1 Outgassing Characteristics of Vacuum Space Materials

The transfer line components inside the vacuum space which could contribute to outgassing are: the fluorocarbon plastic spacers, aluminum foil, glass fiber paper, aluminized Mylar, and the 36% nickel and stainless steel inner and outer lines. Each of these will be evaluated for outgassing characteristics and an attempt will be made to determine the type of gas and the outgassing rate from each material.

5.1.1 Fluorocarbon Plastic Spacers

The transfer line specifications call for six 25% glass-filled fluorocarbon plastic spacers with a total outgassing area of 37 cm^2 . Current high vacuum practice is to vacuum-bake all plastic vacuum space components before installation. The transfer line sections at launch complex 39-B were baked after fabrication, and it is reasonable to assume that the spacers were baked by both manufacturers before fabrication. The outgassing rate for nonbaked fluorocarbon plastic is lower than most other plastics. Santeler [1958] found the outgassing rate of tetrafluoroethylene that had been given a 24-hour nitrogen purge to be 1×10^{-8} torr-liters/ cm^2 -sec after ≈ 5 hours of outgassing at room temperature and up to $\approx 60^\circ \text{C}$.

Santeler did not identify the outgassing constituents. Stanford Research Institute [1966] identified the substances released from fluoroethylene-propylene (500-A) which was vacuum-baked at 125°C and 10^{-6} torr. The major outgassing components were dioctylphthalate and C_{30} terpene oil. The minor components were water and carbon dioxide. Thieme [1961] found the outgassing rate of tetrafluoroethylene to be 2×10^{-8} torr-liters/cm²-sec after being pumped at room temperature for 23 hours, with no previous elevated temperature bakeout. Thieme determined that the principal gases evolved were nitrogen and carbon monoxide, with traces of carbon dioxide and oxygen.

From the above work, it appears as if the outgassing rate of tetrafluoroethylene (TFE) or fluorinated ethylene-propylene (FEP), which has not been given a high temperature bakeout (200-300 °C), is $\approx 10^{-8}$ torr-liter/cm² - sec. A high temperature bakeout would reduce this at least an order of magnitude. It appears as if the major evolved gases are dioctylphthalate, C_{30} terpene oil, nitrogen, and carbon monoxide. All of these gases have a vapor pressure lower than 10^{-11} torr at 20 K, or are easily sorbed at 90 K, and thus present no problem in a transfer line application. In view of the low outgassing rate of fluorocarbon plastic, the small outgassing area, and the type of gases evolved, the outgassing due to the glass-filled fluorocarbon spacers inside the vacuum spaces at KSC will be considered negligible.

5.1.2 Glass Fiber Paper

Reid [1968] conducted some outgassing tests on various metal foils and multi-layer insulation spacer materials. He conducted four tests on glass fiber paper which was processed at 1000°F. Two tests were conducted with a 17-hour vacuum-bakeout at 0.3 microns and 1000°F, and two more tests were conducted with a 17-hour vacuum-bakeout at 0.3 microns and 200°F. After the 17-hour vacuum-bakeouts,

the temperature was decreased 100 degrees to 900 and 100°F, respectively, while pumping for one-half hour. The pressure rise was then monitored for the next 8 hours. For all four of the above tests, Reid recorded a decrease in pressure, and he assigned a zero offgassing rate to glass fiber paper given either of the tests above. The paper actually becomes a fairly good sorbent for moisture after being vacuum-baked. It is reasonable to assume that the glass paper in the lines at KSC would contain moisture and other contaminants after fabrication, but most of this should have been removed during the initial pumpout, especially if the lines were pumped at elevated temperatures. For this evaluation, the outgassing load from the glass paper will be considered to be zero.

5.1.3 Aluminized Mylar

To date, no data have been found giving the outgassing rate of aluminized or plain Mylar after it has been baked near 135°C for a period of 3 - 4 days. This is the vacuum-bakeout treatment given the LH₂ lines at launch complex 39-B, which are the only lines containing the aluminized Mylar multi-layer insulation. The outgassing characteristics of multi-layer insulation materials are very important because of the large surface area involved.

It appears to be the general belief among people who are knowledgeable in vacuum technology that Mylar would tend to outgas slightly more than aluminum foil over a period of years inside a vacuum space, mainly because Mylar is a plastic. However, there is no applicable, comparative outgassing data to confirm this belief. There are several sources of gas to consider when comparing plastic film to metal foil:

- 1) The gases adsorbed on the surfaces, 2) the gases that diffuse to the surface from within the material, hydrogen in the aluminum foil, and various other gases in the case of plastic films, and 3) any chemical

decomposition of the parent material that might occur over a long period of time and give off volatile products. It appears as if the chemical decomposition may be the significant difference between the outgassing characteristics of Mylar and aluminum foil.

Santeler [1958] was one of the first investigators to measure the outgassing rate of Mylar film. His tests were conducted on 0.003-inch thick nonaluminized samples of Mylar A and Mylar V-200, and the duration of the longest tests was only 150 minutes. For one test in which the Mylar was heated, the outgassing rate decreased to 2×10^{-7} torr-liters/cm²-sec after 2 hours. During this test, the sample was exposed to high humidity for 24 hours before evacuation. The sample chamber was evacuated for 20 minutes and the heat was applied at the rate of 20 centigrade degrees/hr. This amounts to $\approx 60^\circ\text{C}$ after 2 hours, which is not representative of the vacuum-bakeout given the vacuum spaces containing Mylar at KSC. Santeler found that a close relationship exists between the slope of the outgassing curve and the sample thickness. He found that the outgassing curves have a slope dependence of $T^{0.5}$ (suggestive of diffusion), and after a relatively short period of time, the slope changes significantly, and a knee develops in the curve. Santeler concluded that once the diffusion gradient penetrates to the center of the sample (sample exposed to vacuum on both sides), a much slower outgassing rate begins. This suggests that thin foil such as aluminum and Mylar in a vacuum space should experience a significant reduction in the outgassing rate after the diffusion gradient penetrates to the center of the foil. When discussing the outgassing characteristics of Mylar film with Santeler [1970], he indicated that, in spite of a good vacuum-bakeout, one could expect trace decomposition of the Mylar film in a vacuum space over long periods of time (3 - 5 years). He indicated that the outgassing rate may be very low, but these decomposition gases would act as a continuous source.

Rivera [1962] conducted some weight loss tests on Mylar A at room temperature, with pressures in the 10^{-8} torr range, over a period of 560 hours. His sample was 0.013 inches thick. He stated that, "this material underwent a rapid diffusion depletion of gases within approximately six hours, with relatively little gas transfer up to the end of this test (560 hours)." Rivera's work appears to substantiate Santeler's conclusion that the outgassing decreases significantly once the diffusion gradient reaches the center of the sample.

Heffelfinger [1970], of the Film Division of E. I. duPont Co., indicates that Mylar does tend to adsorb a small amount of water on the surface because of polar groups on the Mylar film. Heffelfinger indicates that when Mylar is vacuum-baked at 138 - 140°C, linear and cyclic ligamers begin to sublime from the film. At slightly higher temperatures, ethylene glycol is formed as a solid-phase polymerization takes place. He indicates that in outgassing tests he has conducted, in which the Mylar was given an initial bakeout at 138 - 140°C, he observed no difference in the outgassing rate of the clean glass chamber and the same chamber containing the Mylar samples.

Crawford [1967] conducted some outgassing tests on 0.00025-inch thick aluminized Mylar. In this qualitative assessment, he found that the principal outgas constituents of the "as received" foil were N_2 , O_2 , CO_2 , and H_2O . He found that most of the water was desorbed at pressures higher than 5×10^{-3} torr, and he observed that the principal gas to evolve in the 10^{-3} to 10^{-4} torr range was nitrogen. All of the tests were conducted at ambient temperature and no outgassing rates were given.

The Stanford Research Institute [1966] conducted some weight loss measurements on Mylar 500A at 125°C and 10^{-6} torr. The sample

thickness was 0.005 inches. They determined that the major identified substances to be released were low-molecular-weight polyether glycols, and the minor substances released were water and a plasticizer called dioctylphthalate. Identification of the substances was made with a mass spectrometer. They found the weight loss of Mylar 500A, 100S, and 100T to be about the same (0.22% total weight loss), indicating that there is not too much difference in the outgassing characteristics of these three types of Mylar film. The outgassing rates of the samples were not measured.

From the above work cited by various investigators and conversations with people knowledgeable in vacuum technology, the following conclusions about the outgassing characteristics of Mylar can be made.

- 1) Mylar film appears to be slightly susceptible to surface contamination, especially water. This is probably because of polar groups on the surface of the film.
- 2) In the temperature range from 125 - 140°C, the principal substances released are low molecular weight polyether glycols, and the minor substances are water and dioctylphthalate.
- 3) Hydrogen does not appear to be a major or minor outgas constituent during ambient or elevated temperature (125 - 140°C) vacuum-bakeouts.
- 4) A vacuum-bakeout near 135°C appears advisable for a vacuum space containing Mylar. Such a bakeout should remove the surface contamination, the low molecular weight glycols, the dioctylphthalate and water, and possibly a good portion of the hydrogen within the surrounding metal enclosure.

- 5) Mylar film tends to release the major portion of its outgassing substances quickly; probably because it is thin, both sides are exposed to low pressure, and the diffusion gradient soon reaches the center of the film.
- 6) After a good vacuum-bakeout near 125 - 140°C, the outgassing rate is very low. There is no indication what the rate might be, other than $< 10^{-7}$ torr liters/cm² - sec.
- 7) It appears as if there could be a constant trace decomposition of the film over the years. The rate of outgassing contribution from this mechanism is not known and could be negligible.

To determine the outgassing rate of the Mylar film inside the LH₂ vacuum spaces at LC-39 B from the work previously cited is not straightforward. For vacuum spaces that have been baked 3 - 4 days at 125°C, like the LH₂ lines at KSC, the outgassing rate may be near the same order of magnitude as aluminum foil.

In consideration of the foregoing discussion and the fact that the Mylar is extremely thin (0.00025 inches), it appears reasonable to assume that the outgassing rate will be essentially that of the long-term decomposition rate. For the lack of better data, this rate will be chosen as approximately 10 times the outgassing rate of the aluminum foil, or 10^{-12} torr-liters/cm² - sec, and this figure will be used to calculate the total outgassing over a 4-year period. The surface area of the inner line is 113,338 cm², and the surface area of 20 layers of Mylar (both sides) is approximately $2 \times 20 \times 113,338 \text{ cm}^2 = 4,533,520 \text{ cm}^2$. The outgassing rate/year would be

$$4,533,520 \text{ cm}^2 \times \frac{10^{-12} \text{ torr-liters}}{\text{cm}^2 - \text{sec}} \times \frac{3.15 \times 10^7 \text{ sec}}{\text{year}} = 143 \frac{\text{torr-liters}}{\text{year}}.$$

The total outgassing for a 4-year period would be 572 torr-liters.

5.1.4 Stainless and Nickel Steels

Most metals contain a high concentration of hydrogen which results from the manufacturing processes. Stainless steel, and it will be assumed that nickel steel has similar characteristics, has a relatively high diffusion coefficient for hydrogen gas, and the solubility of hydrogen in these steels is also high. These conditions allow hydrogen to slowly diffuse from within the metal to the surface, and then evolve into the vacuum space in a vacuum application.

Some very good experimental work on the outgassing characteristics of aluminum and 300 series stainless steels has recently been completed by Young [1968] and Calder and Lewin [1967]. Both of these investigators agree that the outgassing rate for clean nonbaked stainless steel is on the order of 10^{-10} torr-liters/cm² - sec. They also both agree and have shown experimentally that a hydrogen vacuum-bakeout can reduce the outgassing rate by two orders of magnitude. A hydrogen bakeout is defined as a vacuum-bakeout at 250 - 300°C for 15 - 20 hours. Experimental data from both workers indicate that 99% of the gas evolved during bakeout is hydrogen, and that after bakeout, the hydrogen outgassing rate is reduced to 10^{-12} torr-liters/cm² - sec. and is independent of time. Calder further determined that a vacuum-bakeout at 1000°C for 3 hours reduced the outgassing rate of stainless steel to $\approx 10^{-14}$ torr liters/cm² - sec. The 300 series stainless steel used by Calder contained 0.3 torr-liters of H₂/cm³. The diffusion coefficient for hydrogen in stainless steel at room temperature is $D_r = 5.0 \times 10^{-14}$ cm²/sec [Eschback, 1963], and this increases dramatically

at higher temperatures. The diffusion coefficient at 300°C is $D_{300} = 3.5 \times 10^{-8} \text{ cm}^2/\text{sec}$, and at 1000°C it increases to $D_{1000} = 8.7 \times 10^{-5} \text{ cm}^2/\text{sec}$. These values, and the experimental work above, clearly illustrate the advantage of a high temperature bakeout to reduce the outgassing rate of hydrogen from stainless steel.

Dennison [1970] of Granville-Phillips Co., agrees that the outgassing rate for clean nonbaked stainless steel is $10^{-10} \text{ torr-liters/cm}^2\text{-sec}$, and he adds that this reduces to 10^{-11} after the first year, indicating that the first year is the worst for hydrogen outgassing in a nonbaked system.

The rate of hydrogen outgassing from the metal of a sealed vacuum system or transfer line is very important because hydrogen, like helium and neon, cannot be frozen into a solid with a vapor pressure less than 10^{-11} torr at 20 K, like all of the other atmospheric gases. Hydrogen must be sorbed, or removed by chemical reaction with a getter such as palladium monoxide.

From the above investigations, and the fact that the lines at KSC were not given a good hydrogen vacuum-bakeout, an outgassing rate of $10^{-10} \text{ torr-liters/cm}^2\text{-sec}$ will be chosen for the first year, and $10^{-11} \text{ torr-liters/cm}^2\text{-sec}$ will be chosen for the following years for the stainless and nickel steel inner and outer transfer lines at KSC. The outgassing rate for the steel, exposed to the vacuum space of a typical 10-inch LH_2 spool section 45 feet long, will be calculated. The vacuum space surface area of the inner and outer line is $243,870 \text{ cm}^2$, and it will be assumed that the outgassing characteristics of 36% nickel steel are similar to those of stainless steel (304 ss contains 8 - 12% nickel). For an outgassing rate of $10^{-10} \text{ torr-liters/cm}^2\text{-sec}$, the outgassing load for the first year for the nonhydrogen-baked system is 768 torr-liters. The outgassing load for each following year

at a rate of 10^{-11} torr-liters/cm² - sec, is an order of magnitude less at 77 torr-liters. The total outgassing for a nonhydrogen-baked system over a 4-year period would be ≈ 1000 torr-liters. To point out the advantage of a hydrogen vacuum-bakeout, a similar calculation was made for a baked system having an outgassing rate of 10^{-12} torr-liters/cm² - sec. The outgassing load per year is 8 torr-liters or 32 torr-liters for the entire 4-year period.

5.1.5 Aluminum Foil

The outgassing rate of aluminum foil is very important because there is a considerable amount of foil with large surface area within the vacuum space. Also, it will be shown that the major gas given up by the metal (excluding surface contamination) is hydrogen. Some recent work by Reid [1968] appears to be the only experimental work on the outgassing rate of very thin aluminum foil. Reid conducted four tests on aluminum foil of 0.00025 or 0.0005 inches thickness (The exact thickness of the aluminum foil was not given). The first sample was vacuum-baked for 17 hours at 0.3 microns and 200°F. The sample was then allowed to cool to 100°F for one-half hour while pumping continued. Pumping was stopped and a pressure rise test was then conducted for 8 hours at 100°F, and the gas composition was measured after the 8-hour period. Reid recorded a pressure decrease for the first test. The gas composition within the sample chamber after the 8-hour period was sampled and found to be 41% H₂, 54% water, and 4.8% N₂ or CO. A second similar test on the same sample (34 hours total time at 200°F) also showed a negative pressure change. The chamber gas composition for this test was 57.5% H₂, 35% H₂O, and 7.2% N₂ or CO, showing a significant decrease in water composition. Because of the decrease in pressure, both of these tests were repeated, one of the repeated tests giving increased pressure and the other giving a decreased pressure.

Reid assigned a zero outgassing rate to aluminum foil for these test conditions.

Tests 3 and 4 were conducted at an oven temperature of 1000°F for 17 and 34 hours, respectively, and a decreased temperature of 900°F during the 8-hour pressure rise test. Reid measured an outgassing rate of 7×10^{-12} torr liters/cm² - sec for the 8-hour interval at 900°F, and the gas composition for both of these tests was more than 99% hydrogen. From these tests, one can conclude that a bakeout temperature higher than 200°F is necessary to remove the water adsorbed on the foil, and that the major gas diffusing from within the aluminum is hydrogen.

Deciding upon a value for the outgassing rate of aluminum foil inside a vacuum space over a period of years, from the above tests, is not straightforward. The long term (2 - 5 years) outgassing rate certainly wouldn't be zero. The 1000°F tests indicate there is still hydrogen in the aluminum foil which would undoubtedly diffuse into the vacuum space over the years. The outgassing rate at 900°F cannot be used as a room temperature value because the diffusion coefficient is very temperature dependent. Eschback [1963] found that the diffusion coefficient of hydrogen in stainless steel increased five orders of magnitude from room temperature to 300°C (no measurements were made on aluminum).

Young [1968] measured the outgassing rate of a 0.125-inch thick sample of 1100 type aluminum. The sample was cleaned with detergent and rinsed with acetone. The sample was vacuum-baked at 250°C for 15 hours. The outgassing rate after 24 hours at room temperature was 4×10^{-13} torr-liters/cm² - sec. Similar tests on 304 stainless steel in the same apparatus gave outgassing rates of 10^{-12} torr-liters/cm² - sec for the steel. Young's values for stainless steel are also in agreement with those of Calder and Lewin.

Deciding upon a long term outgassing rate for the aluminum foil inside the vacuum spaces, from the above experimental data, is rather arbitrary. The liquid oxygen transfer lines at LC-39B were vacuum-baked for 3 - 4 days at 150 - 200°C, and it will be assumed that all of the vacuum insulated transfer lines at LC-39A were baked in a similar manner. Considering all aspects of the experimental work by Reid and Young, a value of 10^{-13} torr-liters/cm² - sec will be chosen for the outgassing rate of the aluminum foil (total surface area considered) in the transfer line vacuum spaces at KSC. It was assumed that there are 20 layers of aluminum foil inside the LH₂ vacuum spaces at LC-39A, giving an area of 4,533,520 cm². The outgassing rate would be 14 torr-liters/year or 56 torr-liters for a 4-year period.

5.1.6 Outgassing of a 10-Inch LH₂ Vacuum Space

The total outgassing load from the steel tubing and the aluminum foil or Mylar film for a 4-year period will be calculated, and the resulting pressure rise in the 10-inch vacuum space will be determined. For a vacuum space containing Mylar film, the outgassing load for a 4-year period would be 572 torr-liters for the Mylar and 1000 torr-liters for the steel tubing. The 10-inch spool sections have a vacuum space volume of 254 liters (9 ft³), and the resulting pressure at the end of the 4-year period would be $\frac{1572 \text{ torr-liters}}{254 \text{ liters}} = 6.19 \text{ torr} = 6190 \text{ microns}$. For a vacuum space containing aluminum foil, the outgassing rate for the aluminum foil for a 4-year period would be 56 torr-liters, plus 1000 torr-liters for the steel tubing. The resulting pressure at the end of the 4-year period would be $\frac{1056 \text{ torr-liters}}{256 \text{ liters}} = 4.13 \text{ torr} = 4130 \text{ microns}$. The vacuum space containing Mylar should have residual gas containing $\approx 64\%$ hydrogen, if it is assumed that the metal tubing outgasses hydrogen only, and the Mylar outgasses no hydrogen. The vacuum spaces containing aluminum foil should have residual gas which is mostly hydrogen,

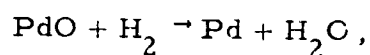
neglecting the effect of palladium monoxide. The calculated outgassing values determined above will be compared to the actual vacuum maintenance data of the vacuum sections at KSC in section 5.3.

It should be pointed out that outgassing calculations are not precise. The calculated pressure rise of 4 and 6 torr over a 4-year period is subject to many variables, such as the metallurgical history of the nickel and stainless steels, the high vacuum fabrication techniques, the presence of sorbents such as charcoal and molecular sieves, and the presence of a getter such as palladium monoxide. There is very little known about the first two variables. Both sorbents, charcoal and molecular sieve, sorb very little hydrogen at low pressures and ambient temperatures, as discussed in section 7. The presence of an active hydrogen getter such as palladium monoxide would tend to convert a good portion of the hydrogen gas into water which would be readily sorbed by molecular sieves, thus reducing the pressure rise due to outgassing. The characteristics of palladium monoxide will be discussed in the following section.

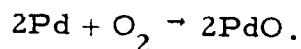
5.2 Palladium Monoxide

A palladium monoxide composition, which is actually 30 - 35% pure palladium monoxide, appears to be the most popular hydrogen getter for use in sealed vacuum systems at the present time and is used by all of the major vacuum-insulated transfer line manufacturers. The primary purpose of using palladium monoxide (PdO) is to convert hydrogen gas, due to metal outgassing, into water, which is then strongly sorbed by a desiccant such as molecular sieves. There is not an abundance of information in the open literature concerning palladium monoxide. The reaction between palladium monoxide and hydrogen gas is a chemical reaction, and because of this, it is preferable that the PdO be in powder or granulated form so that a large surface area is exposed inside the vacuum space.

The primary chemical reaction is [Matsch, 1963]



and further experiments by Matsch have shown that oxygen is also removed from the vacuum space by a secondary reaction,



It should be stressed that water is produced by the primary reaction, and a desiccant such as molecular sieves, which very strongly sorbs water at low pressures, should be used with PdO to sorb the water.

Palladium monoxide is apparently fairly stable in the open atmosphere. It requires no special care, pretreatment, or activation to keep it chemically active prior to use. The hydrogen concentration in the open atmosphere is only 0.5 ppm by volume, as shown in table 7-1. However, there is reason to believe that some chemicals might tend to deactivate (poison) the PdO. These chemicals are sulfur compounds (sulfides) and phosphate compounds (phosphides), none of which are normally encountered in transfer line applications.

Palladium monoxide was placed inside the vacuum spaces of all the 6-inch LOX transfer lines and the 10-inch LH₂ lines at Launch Complex 39A. The 10-inch LH₂ transfer lines at Launch Complex B with charcoal sorbent, are the only lines that do not contain PdO inside the vacuum spaces.

The capacity of PdO to remove hydrogen gas from a vacuum space is important for design purposes. The reaction is temperature dependent, and of course, the pressure in most transfer line applications is less than one torr. Dr. Cohn of Englehard Industries [1970], a commercial supplier of palladium monoxide, points out that the

palladium monoxide which they market is not pure palladium monoxide, but is actually a composition containing $\approx 30 - 35\%$ pure palladium monoxide, and the remainder is a high area, inert carrier material. The PdO composition is sold in powdered or granular form. Cohn indicates that an active composition of their PdO will chemisorb a minimum of 43 torr-liters of hydrogen per gram of palladium monoxide at a temperature of 0°C . Matsch and his co-workers [1963] have experimentally determined that an unspecified palladium monoxide composition will chemisorb 110 torr-liters of hydrogen per gram of PdO at ambient temperatures. Matsch does not give any values for the purity of the PdO composition used in his measurements.

Using an average value of 75 torr-liters of hydrogen/gm PdO, a calculation was made to determine how much PdO would be required to chemisorb the 4-year outgassing load of 1000 torr-liters of hydrogen gas from the nonbaked vacuum space described in the preceding section,

$$\frac{10^3 \text{ torr-liters H}_2}{75 \text{ torr-liters H}_2/\text{gm PdO}} = 13.3 \text{ gms PdO.}$$

The exact amount of PdO inside the vacuum spaces of the transfer lines at KSC is not known, but a fairly good assumption would be that the amount is adequate for the first 5 years.

The amount of palladium monoxide required to convert the hydrogen outgassing load per year beyond the initial years is of interest. The average outgassing load for a liquid hydrogen vacuum space containing aluminum foil multi-layer, after the first year, is $77 + 14 = 91$ torr-liters. The hydrogen capacity of palladium monoxide is ≈ 75 torr-liters/gram. If total conversion is assumed, the amount of palladium monoxide required per year to take care of the hydrogen outgassing would be

$$\frac{91 \text{ torr-liters/year}}{75 \text{ torr-liters/gram}} = 1.2 \text{ grams/year.}$$

If it is assumed that the initial charge of palladium monoxide in the vacuum spaces at KSC was sufficient to last 5 years, calculations indicate that ≈ 1.2 grams PdO per vacuum space per year should be sufficient to convert the hydrogen for the following years.

A novel technique of placing a fresh supply of PdO inside the vacuum space without disturbing the vacuum has been patented by Matsch [1963]. This is accomplished by placing one or more glass capsules full of PdO inside a soft copper tube attached to the warm wall of the vacuum space. At some later date when a fresh supply of PdO is required, the soft copper tube may be deformed so as to break the glass capsule inside and release a fresh supply of PdO into the vacuum space.

Palladium monoxide is fairly expensive at \$1.25/gm when purchased in quantities of at least 1000 grams. However, it has very high capacity for hydrogen, requires very little special care in handling, and when combined with molecular sieves, appears to be a very effective answer to long term hydrogen outgassing.

5.3 Vacuum Maintenance Study

The present procedure for checking the pressure inside the vacuum sections of LC-39A using the thermocouple meter and probe, is to connect and zero the meter, record the pressure inside the thermocouple probe space (the volume between the closed bellows type valve and the 1-inch pipe plug), open the valve to the main vacuum space, and record the pressure for that particular vacuum section. The maintenance records indicate that, more often than not, the pressure inside the probe space is greater than 1000 microns (possibly atmospheric). The exact pressure is difficult to determine because thermo-

couple meters are not accurate near full scale. The pressure buildup in this space is most likely due to leakage around the thermocouple probe threads, the larger threads within the "Tee", leakage around the thermocouple probe pins, or a leak in the valve bellows. In any case, a leakage problem exists with a good portion of those probe spaces, and the present procedure of dumping the air within the probe space into the main vacuum space everytime the vacuum section is checked is not consistent with good high vacuum practice. A calculation will better show why this is not to be recommended.

The probe space has a volume of $\approx 4 \text{ in.}^3$, and the volume of the vacuum space for a 10-inch spool piece 45 feet long is $\approx 15,600 \text{ in.}^3$. Assume the initial pressure in the vacuum space is zero and the pressure in the probe space is one atmosphere (worst condition). Opening the valve would raise the pressure in the main vacuum space to

$$P = 760 \text{ torr} \times \frac{4 \text{ in.}^3}{15,600 \text{ in.}^3} = 0.195 \text{ torr} \approx 200 \text{ microns.}$$

If the probe space pressure were 0.1 atmosphere, the pressure rise would be 20 microns. In reality, the pressure in the probe space is probably much less than one atmosphere, and it is only conjecture to say what the pressure is when the thermocouple gauge reads beyond full scale (1 or 5 torr). However, the important point is there is a good portion of these units experiencing leakage, and it is not inconceivable that a few of these spaces are reaching pressures near one atmosphere. A condition exists wherein a very good vacuum space could experience a pressure rise of $12 \times 200 = 2400 \text{ microns/year}$ because of a leak into the probe space, assuming the vacuum spaces are checked monthly.

The situation is even more serious for a 6-inch LOX vacuum space because the vacuum space volume is less, 10,584 in.³ compared to 15,600 in.³ for the hydrogen spool sections. Assuming the same 4 in.³ volume for the probe space, there would be $\approx 32\%$ more pressure rise (3168 microns/year) in the LOX vacuum space because of the smaller volume.

Moreover, the sorbents in the vacuum spaces would sorb very little air at ambient temperatures. Both charcoal and molecular sieve have very little sorption capacity for nitrogen and oxygen at these temperatures and pressures, although the molecular sieves would sorb most of the moisture and CO₂.

Considering the above calculations, one must conclude that the present practice of dumping the probe space leakage into the main vacuum space is not good high vacuum procedure, and this space should be evacuated before the valve is opened. Also, there are indications in the vacuum maintenance data that the bellows type valves between the probe space and main vacuum space have been inadvertently left open on several occasions, after checking the vacuum section. The vacuum checking procedure should be amended to double check against this possibility.

The vacuum maintenance data for the liquid hydrogen lines of LC-39A, covering the period from May 1966 to July 1969 (38 months), are plotted in figure 5-1. Each of the 56 vacuum spaces was checked monthly as normal vacuum maintenance procedure, and the space was pumped if the pressure exceeded the upper limit established at that time (400 or 1000 microns). The identification number of the vacuum space is along the abscissa, and the time, starting at May 1966, is along the ordinate. A dot indicates the section was vacuum pumped at that time.

VACUUM MAINTENANCE DATA LH₂ Vacuum Insulated Lines

LC - 39A

5-66 to 7-69

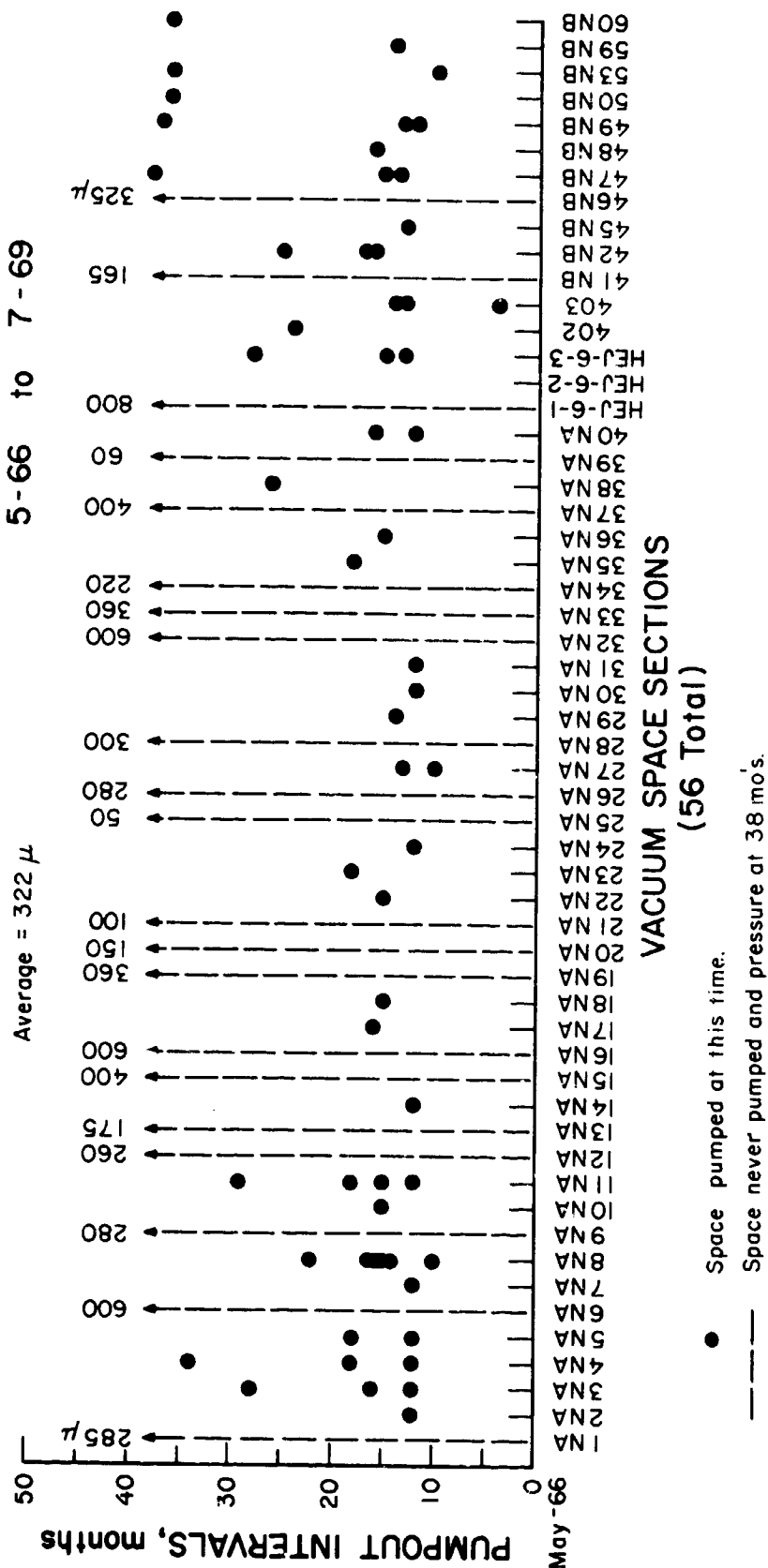


Figure 5-1 Vacuum Maintenance Data for the LH₂ Vacuum Spaces of LC-39A.

Dashed lines with a pressure reading at 38 months indicate that the vacuum space was never pumped during the 38-month period, and the pressure in the vacuum space at 38 months is that pressure written on the plot.

The data show that there are 21 very good vacuum sections with a pressure rise which averages 322 microns for a 38-month period. Thus, 38% of the sections are very good with an average pressure rise of ≈ 100 microns/year. Eighteen of the sections were pumped only once during the 38-month period. This represents 32% of the total, and vacuum pumping once during a 38-month period is not unreasonable. Nine of the sections (16%) were pumped two or three times during the same period. These sections probably have small leaks. Only two sections (3%) were pumped four or more times. These sections are 8NA and 11NA. They obviously have a leak and should be repaired. This is a fairly small percentage requiring extensive maintenance over a 38-month period.

The vacuum maintenance data for the cross-country liquid oxygen transfer lines at LC-39A were not plotted. Most of these sections were pumped fairly soon after installation, and for some reason, the vacuum integrity of these sections is considerably worse than any of the other vacuum insulated transfer lines. It was felt that little additional information could be gained from a plot of this data.

The vacuum maintenance data for the cross-country liquid oxygen lines of LC-39B were plotted in a similar manner, as shown in figure 5-2. These vacuum sections contain aluminum foil and glass fiber paper insulation, molecular sieve sorbent, palladium monoxide hydrogen getter and were initially sealed in March, 1967 [Martindale, 1970]. The joint covers are sealed permanently by welding, and the spool sections were vacuum-baked after fabrication. The vacuum maintenance

VACUUM MAINTENANCE DATA

LOX Vacuum Insulated Lines

LC - 39 B

3-67 to 4-70

These Vacuum Spaces Initially
Sealed \approx March, 67.

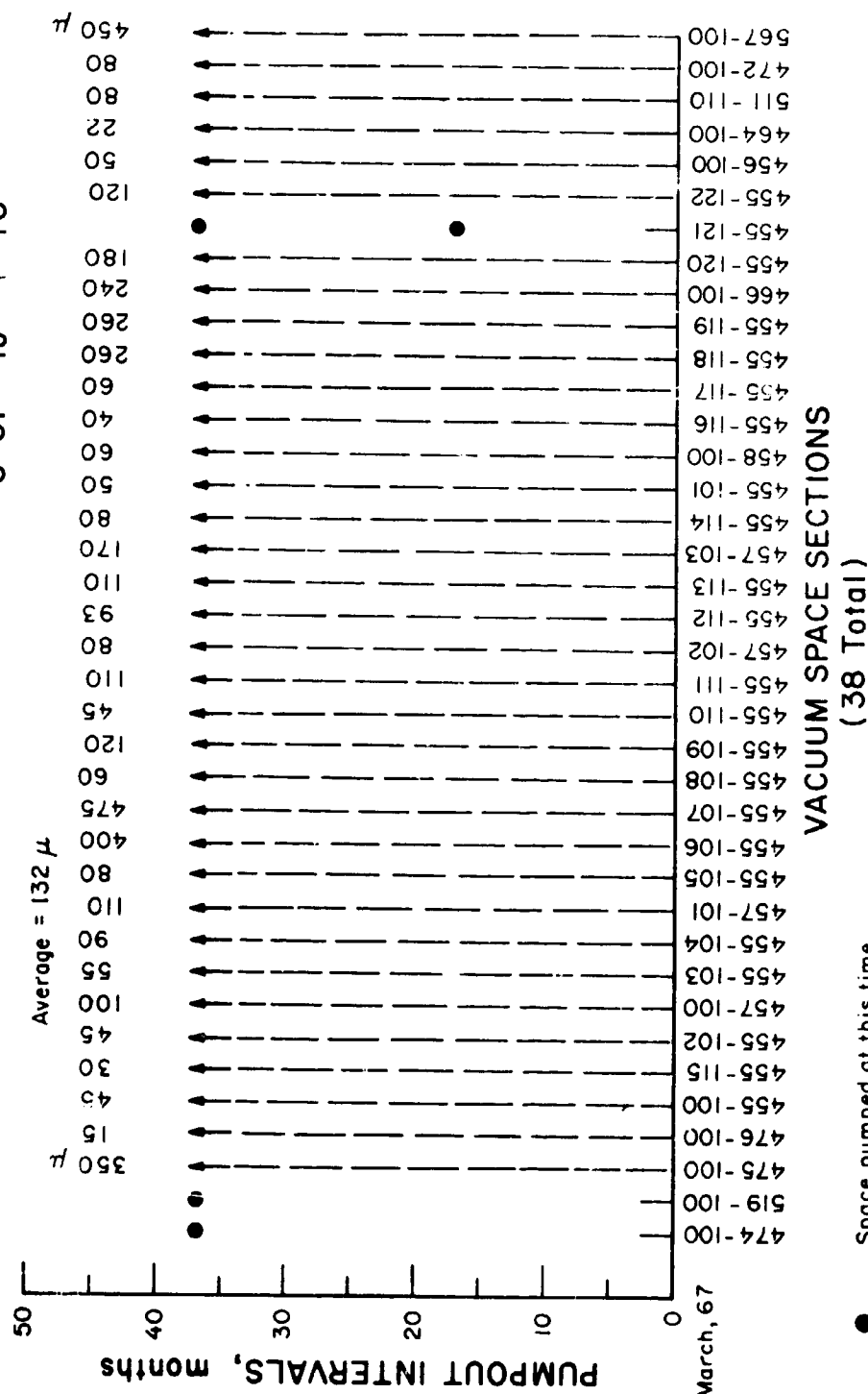


Figure 5-2 Vacuum Maintenance Data for the LOX Vacuum Spaces of LC-39B.

data for this section of line indicate that most all of the vacuum spaces are very well sealed, and the performance of these sections is considerably better than the LH_2 transfer line sections of LC-39A. Of the 38 sections considered in this transfer line, only three indicate leakage, (474 - 100, 519 - 100, and 455 - 121). The other 35 sections have evidently not been pumped and have an average vacuum level of 132 microns after 40 months of service (to April 1970), giving an average pressure rise of 3.3 microns per month. Initially, these lines were pumped to ≈ 1 micron by the manufacturer and indicated less than 8 microns per day pressure rise for the acceptance test [Martindale, 1970]. This is exceptionally good performance and probably represents the state of the art for vacuum insulated transfer lines in a field installation of this size.

The vacuum maintenance data for the cross-country liquid hydrogen lines of LC-39B were also plotted. The data are shown in figure 5-3. These vacuum sections contain aluminized Mylar multi-layer insulation and charcoal sorbent. These vacuum spaces contain no palladium monoxide, and the joint covers are also sealed permanently by welding. The vacuum spaces were sealed about November 1966 with \approx one micron pressure [Martindale, 1970]. These vacuum spaces show very good performance. There is no record of any of the spaces being pumped during the first 39 months. Twenty-three of the sections were pumped during the 39 to 46-month period. The remaining 24 have never been pumped and have an average pressure of 526 microns as of 9 - 70. Only two sections have been pumped more than once, and one of these had a leaking burst disc.

This particular vacuum maintenance data is most interesting because these spaces contain no palladium monoxide; they have Mylar multi-layer insulation, and recent residual gas analyses (RGA) data

VACUUM MAINTENANCE DATA LH₂ Vacuum Insulated Lines LC - 39 B

These Vacuum Spaces Initially
Sealed ≈ Nov., 1966.

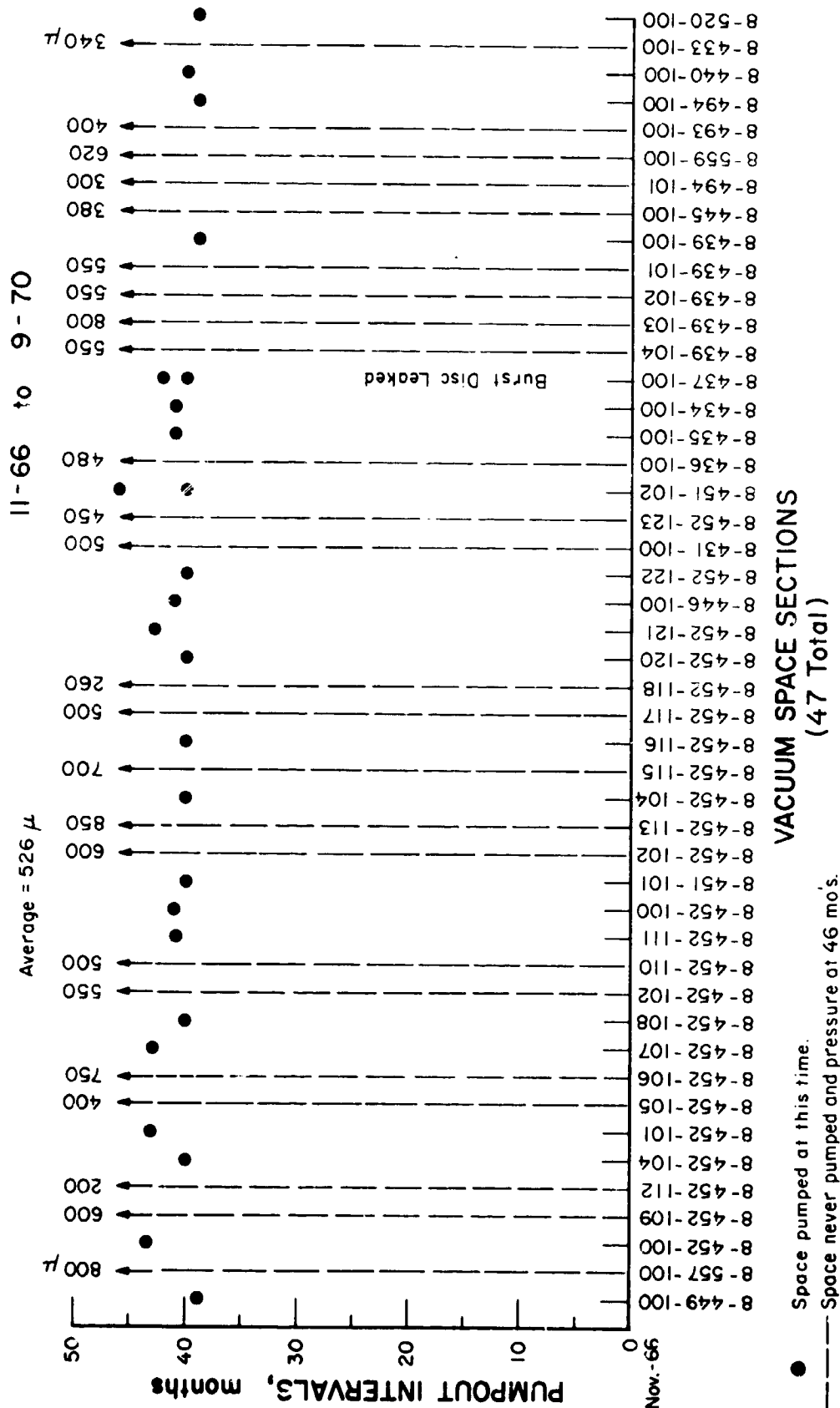


Figure 5-3 Vacuum Maintenance Data for the LH₂ Vacuum Spaces of LC-39B.

have been provided for a good share of the sections that have been recently pumped. The RGA data are presented in table 5-1. RGA data for the other transfer lines were not available or were not provided.

The amount of hydrogen in the vacuum spaces is probably the most interesting aspect of these analyses. The gas composition in some of the vacuum spaces is 80 - 90 percent hydrogen, and most all the spaces show fairly high hydrogen concentration. The high hydrogen concentration is most likely due to the hydrogen outgassing of the nickel and stainless steel. Two of the spaces (8-452-102 and 8-437-100) have ratios for oxygen and nitrogen near that of air, obviously due to atmospheric leakage into the vacuum spaces. Another interesting facet of these analyses is the relatively high concentration of nitrogen (a portion of this could possibly be CO) in most of the spaces. This might possibly be due to the Mylar outgassing, or to residual nitrogen gas which was used for purging and pressurizing prior to initial sealing.

The pressure in these vacuum sections is also of great interest. The average value of 526 microns for the sections that have never been pumped, and the approximate value of 1000 microns for the remaining sections recently pumped, give an overall average of ≈ 750 microns for all 47 sections over the 46-month period. This value is almost an order of magnitude lower than the 6000 microns calculated from outgassing data. The higher calculated figure is probably due to choosing a value too high for the outgassing rate of the steel for the first year. There is also the possibility that a significant amount of the gas is being sorbed by the charcoal, but activated charcoal should sorb very little nitrogen or hydrogen at these partial pressures and temperatures.

These vacuum space pressures tend to indicate that aluminized Mylar does outgas very little after it has been given a good vacuum-bakeout, and that the outgassing rates chosen for the steel, Mylar, and

Table 5-1

Residual Gas Analyses of the
Liquid Hydrogen Vacuum Spaces at LC-39B

Vacuum Section No. and Date	Residual Gas Composition (%)						
	CO ₂	He	H ₂	O ₂	Ar	N ₂	H ₂ O
8-452-100 (6-70)	< 0.5	< 0.5	95	< 0.5	< 0.5	4.0	0.8
8-452-104 (3-70)	< 0.5	----	37	< 0.5	0.6	68.3	----
8-452-101 (6-70)	0	0	80	0	0.8	18.6	0.5
8-452-107 (6-70)	< 0.5	< 0.5	86.9	< 0.5	< 0.8	11.9	----
8-452-111 (4-70)	----	----	49.4	0.7	----	48.1	----
8-451-100 (4-70)	----	----	28.8	0.1	0.9	70.2	----
8-451-101 (3-70)	< 0.5	< 0.5	90.4	< 0.5	0.9	8.7	2.5
8-452-114 (3-70)	< 0.5	----	73.9	< 0.5	0.7	25.4	----
8-452-116 (6-70)	0	0	79.8	0	1.2	19	0
8-452-121 (6-70)	< 0.1	0	90.6	0	0.9	8.1	0.4
8-452-102 (3-70)	< 0.1	< 0.1	39.8	< 0.1	1.7	58.6	< 0.1
Burst Disc Leaking (9-70)	< 0.5	0	0	22	----	77	< 0.5
8-434-100 (4-70)	----	----	50.7	----	1.7	47.6	----
8-437-100 (3-70)	0.1	----	0.3	14.8	1.0	83.8	----
8-440-100 (6-70)	< 0.5	< 0.5	34	< 0.5	0.7	64.6	----

aluminum foil were all conservative, possibly an order of magnitude too high. This is especially true when considering that the probe space pressure is most always greater than 1500 microns, and this gas is dumped into the vacuum space every time the vacuum space pressure is checked. The gas leaking in these probe spaces could account for a significant portion of the pressure increase. However, the high hydrogen composition of most of the vacuum spaces indicates that most of the pressure increase is due to hydrogen outgassing from the metal and not atmospheric leakage from the probe space. These liquid hydrogen transfer line vacuum sections containing Mylar probably represent the best experimental data available on the long term outgassing characteristics of 0.00025-inch thick aluminized Mylar foil. There is a very large area of Mylar and the data cover a period of 46 months.

Also, it is interesting to compare the average pressure of the LOX line vacuum spaces of LC-39B with the average pressure of the LH₂ line vacuum spaces of LC-39B. Both of these transfer lines were manufactured by the same firm and both were given approximately the same vacuum-bakeout. The LH₂ lines were baked at 135°C for 3 - 4 days, and the LOX lines were baked at a slightly higher temperature for the same period of time. The major differences between the two lines (other than size) are the palladium monoxide in the LOX line vacuum spaces and the different multi-layer insulation materials. The average pressure of the 35 LOX vacuum spaces that have never been pumped is 132 microns, and the average pressure of the 24 LH₂ vacuum spaces that have never been pumped is 526 microns. Considering the high concentration of hydrogen in the LH₂ vacuum spaces, the hydrogen chemisorbing ability of the palladium monoxide present in the LOX vacuum spaces, neglecting any sorption capacity of the molecular sieves or charcoal (for gases other than water), and the fact that the

volume/surface area ratio of both spaces is almost the same, it appears as if the aluminized Mylar outgasses at about the same rate as the aluminum foil, and both have very low outgassing rates.

5.4 Conclusions Derived From Transfer Line Evaluation

The main outgassing load in the vacuum spaces containing Mylar multi-layer insulation is hydrogen, and this is most likely true for the vacuum spaces containing aluminum foil and glass fiber spacer material. Because the major outgassing constituent in a vacuum space is hydrogen, it is most important to use palladium monoxide, especially in a liquid oxygen transfer line where the sorption of hydrogen is difficult.

It appears as if the outgassing rate of aluminized Mylar, which has been given a good vacuum-bakeout, is about the same as that of aluminum foil given a similar bakeout, and that both of these have an outgassing rate of $\approx 10^{-12}$ torr-liters/cm² - sec when given a 135°C vacuum-bakeout for 3 - 4 days.

The outgassing constituents of Mylar appear to be condensible at 20 K (glycols, dioctylphthalate, and water), whereas the major gas to diffuse from aluminum foil is hydrogen, which is not condensible. Also, it appears that if there is any trace decomposition of the Mylar film over a long period of time, the outgassing rate due to this decomposition is extremely low.

The calculated total outgassing of a typical 10-inch vacuum space containing Mylar is 1560 torr-liters during 4 years, which would increase the vacuum space pressure to 6 torr during that period if palladium monoxide were not present. The actual pressure rise, measured over a 46-month period, for the 10-inch vacuum sections containing Mylar, averaged ≈ 750 microns. This is almost an order of magnitude less than the 6000 microns calculated. The calculated value could be high for a number of reasons. It is most likely due to choosing

an outgassing rate too high for the steel during the first year. Another possibility is that the charcoal sorbent is sorbing some of the gas at ambient temperatures.

The average chemisorption capacity of palladium monoxide for hydrogen gas, at ambient temperatures and pressures near one micron, is 75 torr-liters (90 cm^3 [STP]) per gram of PdO. A calculation indicates that approximately 13 grams of PdO would be required to convert the 1000 torr-liters of hydrogen gas to water during the 4-year period, and after this period, approximately 1.2 grams of PdO per year should consume the hydrogen outgassing from a typical 10-inch vacuum section.

The present practice of dumping the probe space leakage into the main vacuum space should be discontinued. The probe space should be evacuated with a small portable pump before the valve to the main vacuum space is opened.

The cross-country transfer line vacuum spaces at LC-39B (both oxygen and hydrogen) are exceptionally well sealed. If care is exercised in taking vacuum measurements, these sections should require little vacuum maintenance. A large percentage of the cross-country hydrogen transfer line vacuum space sections at LC-39A are also well sealed. Only 3% have required pumping four or more times. Small leaks appear to exist in approximately 16% of these vacuum sections. The expense of pumping compared to the expense of repairing these small leaks would most likely dictate periodic pumping. Pumping once a year is not an arduous or expensive task. Vacuum space repair is usually extensive, requiring vacuum bakeout upon completion; vacuum spaces with larger leaks, requiring frequent pumpout, obviously must be repaired.

6.0 Effect of Vacuum Space Pressure on System Heat Leak

The purpose of this section is to determine the effect of increased vacuum space pressure on the total heat leak of the LOX and LH_2 systems. The specification heat leak for each system will be used as an approximate base heat leak for that system when there is a good insulating vacuum ($< 10^{-4}$ torr) inside the vacuum spaces. The base heat leak for each system will be defined as Q_0 . The liquid hydrogen and liquid oxygen systems will be considered separately. In each case, the defined system will be the portion of the transfer lines transferring liquid between the storage tank and the flight vehicle. The heat leak due to supports, valves, spacers, radiation, and other components will be considered constant. The heat leak due to residual gas conduction within the vacuum spaces is the only variable to be considered.

6.1 Residual Gas Conduction in Multi-Layer Systems

When considering heat transfer due to residual gas conduction, there are three regimes to the familiar "S" shaped curve [Barron, 1966] of heat transfer vs. pressure shown in figure 6-1. At pressures of ≈ 10 torr and above, the thermal conductivity of a multi-layer system is independent of the pressure, and the heat transfer is very high. In the region from 10 torr down to $\approx 10^{-4}$ torr, the thermal conductivity drops sharply as the "free molecule" conduction region is approached. At pressures below $\approx 10^{-4}$ torr, "free molecule" conduction exists, and the heat transferred by the residual gas is directly proportional to the gas pressure. However, in a multi-layer insulation system, the heat transferred by the residual gas at low pressure is only a small portion of the total heat transferred through the insulation, and the apparent thermal conductivity of the system decreases very slowly at pressures below 10^{-4} and especially below 10^{-5} torr. Because the heat transferred by the residual gas at pressures $< 10^{-4}$ torr is only a small portion of

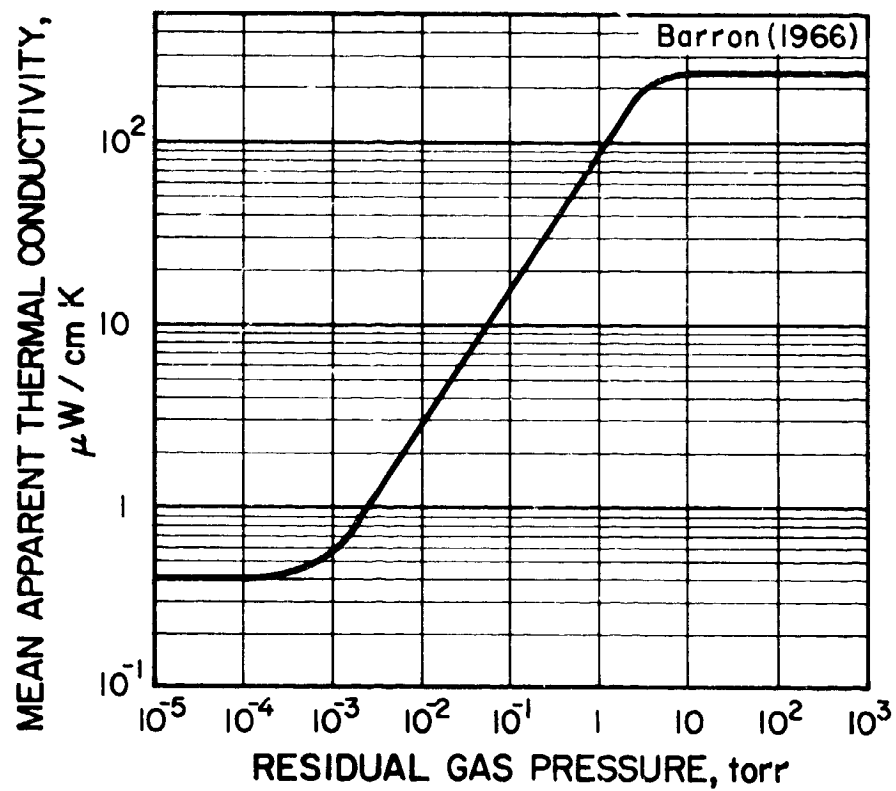


Figure 6-1 Variation of Thermal Conductivity with Residual Gas Pressure for a Typical Multi-Layer Insulation System.

the total heat transferred, the type of residual gas has little effect on the total heat transferred at pressures $< 10^{-4}$ torr. For minimum heat transfer, such as in cryogenic transfer lines, it is most important to maintain vacuum space pressure at or below 10^{-4} torr.

Experimental data, showing the effect of vacuum space pressure on the apparent thermal conductivity of multi-layer systems, were compiled for two temperature ranges. Analytical data were not included. The heat transfer data for the temperature range 293 - 20 K are shown in figure 6-2, and the data for the range 300 - 77 K are shown in figure 6-3. Table 6-1 lists the pertinent parameters of the multi-layer insulation systems. Since the heat transfer in multi-layer systems at pressures < 1 torr, is only slightly dependent upon the thermal conductivity of the gas and the distances between confining surfaces, average values for the apparent thermal conductivity will be taken from the experimental data to determine the effect of increased vacuum space pressure on the total system heat leak of Launch Complex 39.

There are other variables in a multi-layer insulation system which can significantly increase the heat transfer. These are, protrusions through the multi-layer, insulation gaps, insulation joints, and compressive loading. Multi-layer systems are especially sensitive to compressive loading. However, for this analysis, none of the above can be accurately assessed and consequently will not be considered. The transfer lines at KSC have already been fabricated by qualified manufacturers, and the heat transfer attributable to any of the mechanisms above will be considered constant and part of the system heat leak. For this analysis, the effect of increased residual gas pressure on the system heat leak is the only variable under consideration.

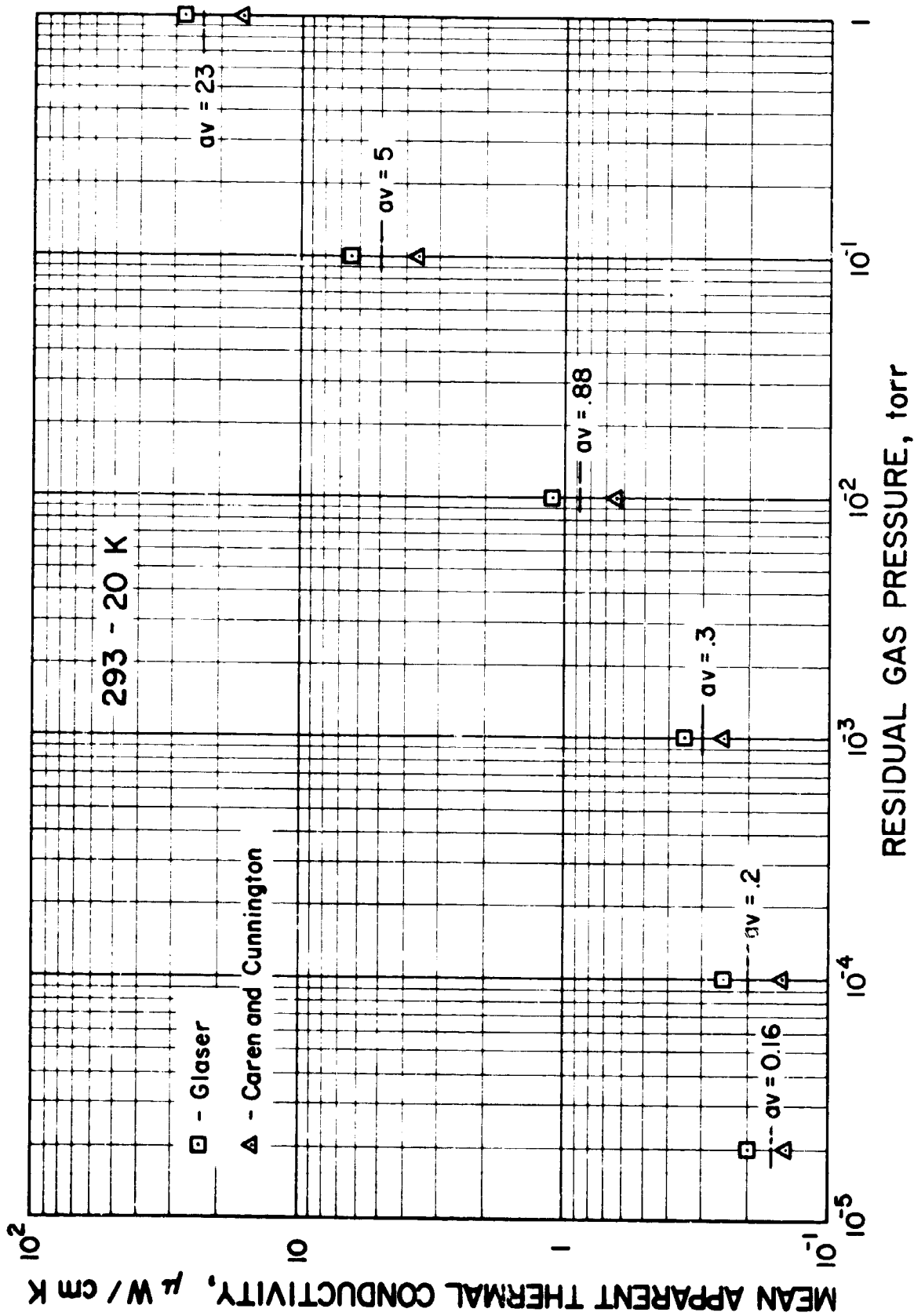


Figure 6-2 Experimental Thermal Conductivity Data for Multi-Layer Insulation Between 293 and 20 K.

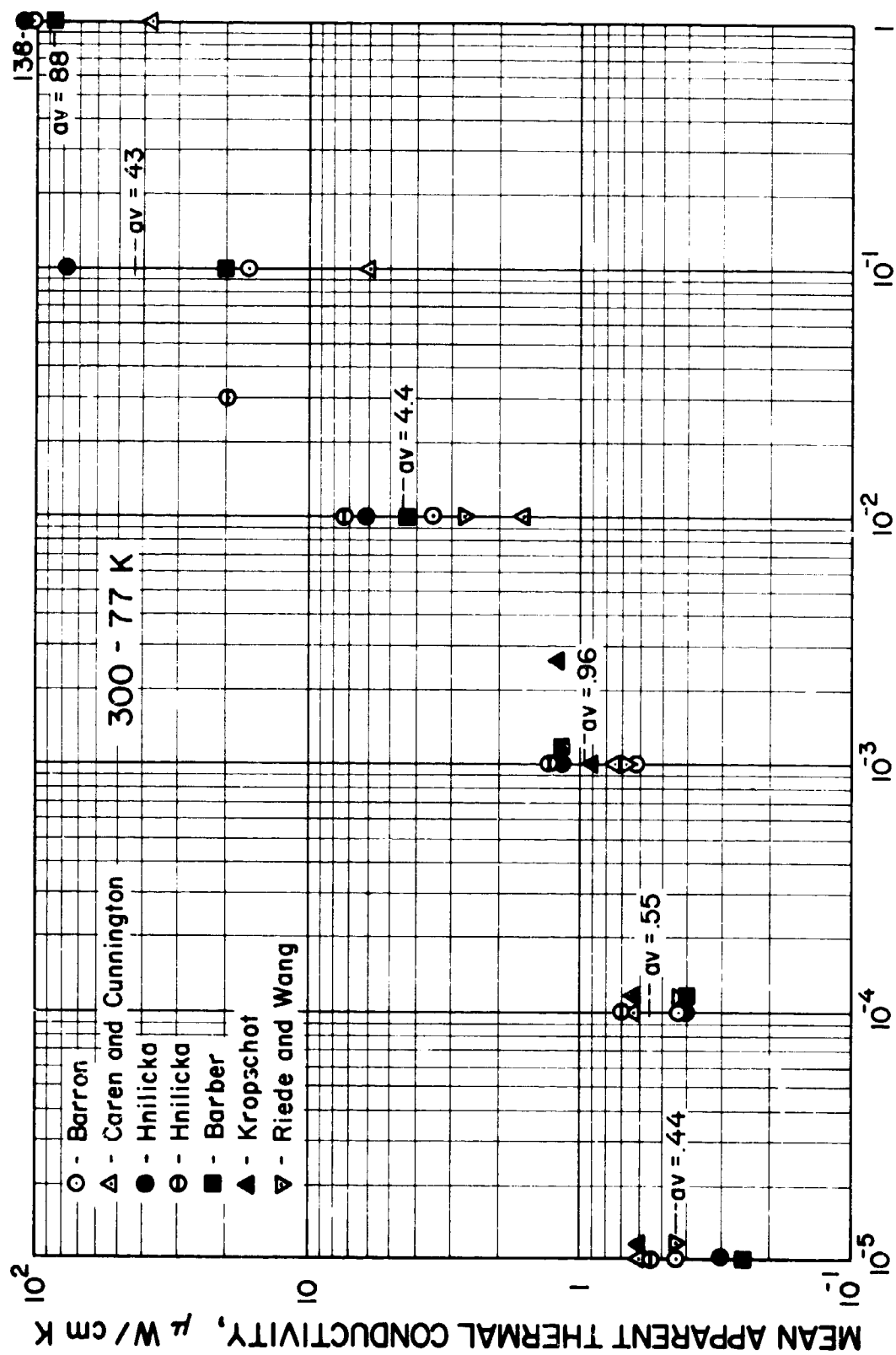


Figure 6-3 Experimental Thermal Conductivity Data for Multi-Layer Insulation Between 300 and 77 K.

Table 6-1
Multi-Layer Experimental Data (300 - 77 K)

Barron (1966)-----	(300 - 90 K), typical multi-layer insulation, 60 layers/in., residual gas not given.
Caren & Cunningham (1967)-----	(300 - 77 K), no information given.
Barber (1966)-----	(300 - 77 K), 10 shields of 0.002 in. thick tempered aluminum, 11 spacers of 1/8 x 1/8 in. silk netting 0.022 in. thick, helium residual gas.
Hnilick (1960) -----	(300 - 77 K), 50 aluminum foil shields with 60 fibrous spacers, nitrogen residual gas.
Hnilicka (1960) -----	(300 - 77 K), 0.00025 in. thick aluminized Mylar, 25 sheets, 0.5 in. thick, nitrogen residual gas.
Kropschot (1961)-----	(300 - 76 K), aluminum foil shields, 0.008 in. thick glass fiber paper, 22 shields/cm, 3.3 cm thick, nitrogen residual gas.
Riede & Wang (1960)-----	(300 - 77 K), glass fiber mat and aluminum foil, 40 - 80 layers/in., residual gas not given.

Multi-Layer Experimental Data (293 - 20 K)

Caren & Cunningham (1967)-----	(293 - 20 K), helium residual gas.
Glaser (1967)-----	(293 - 20 K) aluminum and fiberglass paper, helium residual gas.

6.2 Liquid Hydrogen System (10-Inch V. J. Lines - Complex 39A or B)

The system under consideration is all of the vacuum jacketed transfer lines extending from the LH_2 storage tank to the flight vehicle connections. Specifically, these are HVJ-1A, 1B, and 1C. This is the portion of the system flowing hydrogen when the flight vehicle is being loaded or replenished. Transfer lines HVJ-1D and HVJ-1E are not considered, since they extend from the LH_2 storage tank to the truck supply connections.

The specification heat leak for the system as defined is tabulated below.

HVJ-1A - - - -	8,000 BTU/hr = 2344 Watts
HVJ-1B - - - -	1,000 BTU/hr = 293 Watts
HVJ-1C - - - -	7,000 BTU/hr = 2051 Watts
Total Specification =	<u>16,000 BTU/hr = 4688 Watts</u>
Heat Leak	<u>\approx 4700 Watts</u>

The specification heat leak as given for the three sections of vacuum jacketed line will be considered a good approximate heat leak for the system when there is a pressure of 10^{-4} torr in each vacuum space. This will be defined as $Q_o = 4700$ watts and includes all modes of heat transfer.

The next task is to determine what portion or percentage of this base heat leak (Q_o) is due to residual gas conduction in the vacuum spaces. This will be done by calculating the heat leak due to residual gas conduction, using the total area of all the V. J. lines in the defined system, and an average apparent thermal conductivity for a multi-layer system from the experimental data of figure 6-2.

The heat leak with a pressure of 10^{-5} torr within the vacuum spaces will also be calculated. At this pressure, the heat leak due to

residual gas conduction is negligible and will be considered zero. It will be assumed that all the heat transfer through the vacuum spaces at 10^{-5} torr is due to radiation and conduction through the multi-layer. The heat leak at 10^{-4} torr will then be calculated, and the difference between these two values will be considered to be the heat leak due to residual gas conduction at 10^{-4} torr within the vacuum spaces.

The surface area of the inner lines within the defined LH_2 system was calculated as follows.

$$\begin{aligned}
 1568 \text{ lineal feet of 10-inch V. J. line} &= 4413 \text{ ft}^2 \\
 123 \text{ lineal feet of 8-inch V. J. line} &= 278 \text{ ft}^2 \\
 7 \text{ lineal feet of 4-inch V. J. line} &= 91 \text{ ft}^2 \\
 \hline
 \text{Total Area} &= 4782 \text{ ft}^2 \\
 &= \underline{4442 \times 10^3 \text{ cm}^2}
 \end{aligned}$$

Average values for the apparent thermal conductivity of a multi-layer system will be taken from the experimental data shown in figure 6-2, and the insulation space will be taken as 2 cm thick. At 10^{-5} torr pressure within the vacuum spaces, the apparent thermal conductivity (\bar{k}) = $0.16 \mu \text{ watts/cm} - \text{K}$, and the heat leak to the system through the multi-layer is

$$\begin{aligned}
 Q &= \frac{0.16 \mu \text{ Watts}}{\text{cm} - \text{K}} \times \frac{280 \text{ K}}{2 \text{ cm}} \times 4442 \times 10^3 \text{ cm}^2 \\
 Q &= \underline{100 \text{ Watts}} .
 \end{aligned}$$

It will be assumed that none of this 100 watts is due to residual gas conduction.

At 10^{-4} torr pressure, the apparent thermal conductivity (\bar{k}) is $0.2 \mu \text{ watts/cm} - \text{K}$, and the heat transfer to the system through the vacuum spaces is 124 watts. The difference between these two values

(24 watts) will be considered the heat leak due to residual gas conduction at 10^{-4} torr vacuum space pressure, and the heat leak due to all other modes of heat transfer is $4700 - 24 = 4676$ watts. The percent of total system heat leak due to residual gas conduction within the vacuum spaces is $\frac{24}{4700} = .005 = 1/2\%$. At one micron pressure (10^{-3} torr), the average apparent thermal conductivity is 0.3μ watts/cm - K, and the heat transfer to the system through the vacuum spaces is 200 watts. In this case, the heat transfer due to residual gas conduction is $200 - 100 = 100$ watts. Similar calculations for 10, 100, and 1000 microns pressure give heat transfer values due to residual gas conduction of 483, 3010, and 14,306 watts, respectively. This information is tabulated in table 6-2, showing the ratio of Q/Q_0 and the percent increase in total system heat leak for the various pressure levels.

Table 6-2

Liquid Hydrogen System Heat Leak

vacuum space pressure (microns)	Q (watts) gas cond.	Q (watts) syst, total	Q(BTU/hr) syst, total	Q/Q_0	% increase in total syst. heat leaks
0.1	24	$Q_0 = 4,700^*$	16,040	1	Zero
1	100	4,776	16,300	1.02	1.6%
10	483	5,159	17,608	1.10	9.8%
100	3,010	7,686	26,232	1.64	64 %
1000	14,206	18,906	64,524	4.02	302 %

* Includes 24 watts due to gaseous conduction.

A plot showing the effect of residual gas pressure on the heat leak for the hydrogen system is shown in figure 6-4. The data indicate that vacuum space pressures up to one micron do not cause an appreciable increase in system heat leak, and this pressure level could most likely be tolerated. Interstitial pressures near 10 microns cause a 10 percent

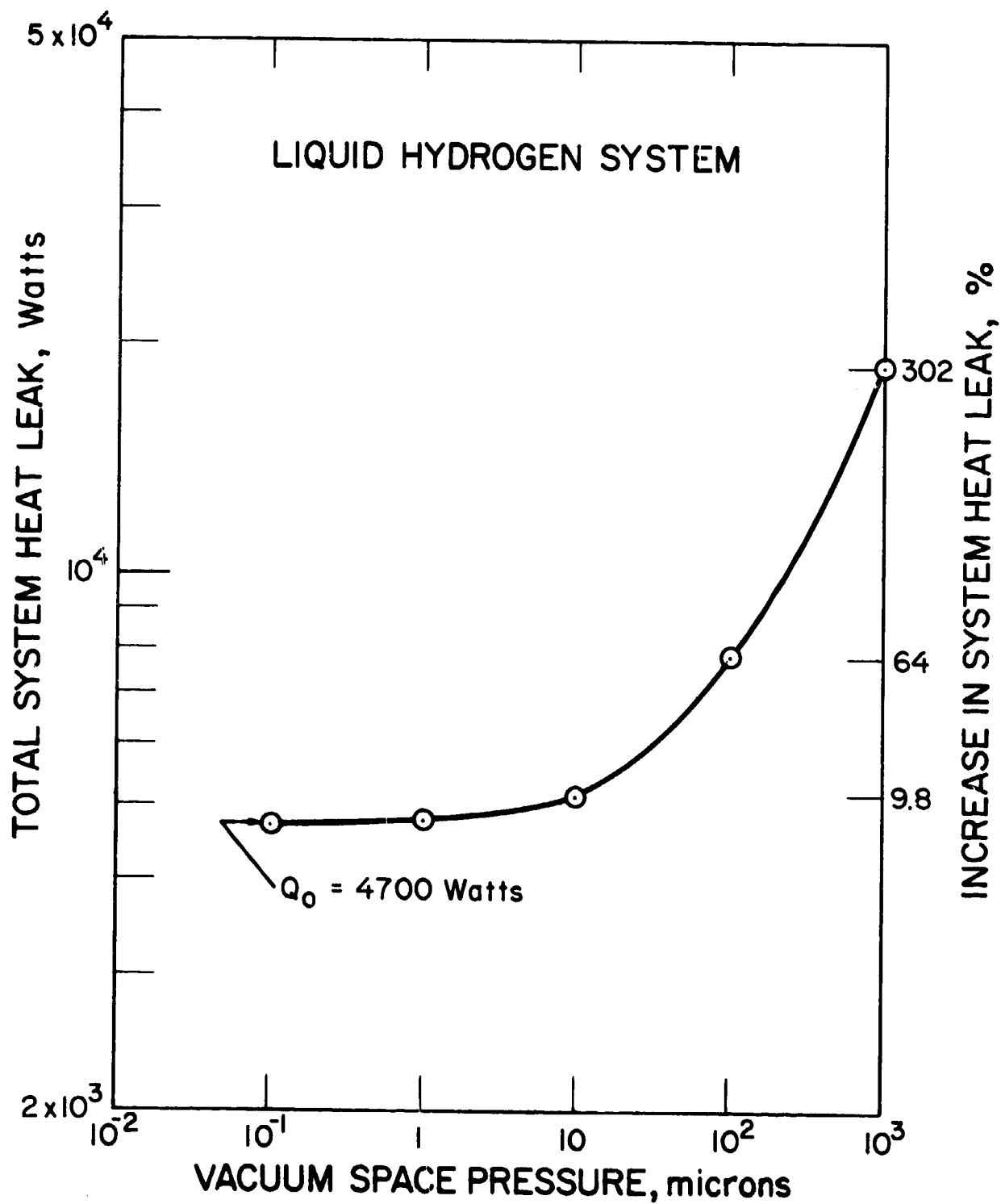


Figure 6-4 Liquid Hydrogen System Heat Leak vs. Vacuum Space Pressure.

increase in system heat leak, and above 10 microns, the heat load increases very rapidly. A similar analysis will be made on the liquid oxygen system.

6.3 Liquid Oxygen System (6-Inch V. J. Lines - Complex 39A or B)

The liquid oxygen system under consideration is defined as follows:

- 1) the 6-inch line extending from the storage tank, across country to the 200-foot level, and ending with V. J. section 57 OD. The specification heat leak for this portion of the system is 12,200 BTU/hr, and
- 2) the launch umbilical tower disconnect piping with a specification heat leak of 1100 BTU/hr, and
- 3) the 8-inch V. J. line at the 120-foot level which includes sections 49 OD, 50 OD, and 51 OD, with a specification heat leak of 1300 BTU/hr, and
- 4) the 10-inch V. J. line at the 30-foot level, which includes sections 43 OB, 44 OB, and 45 OB with a specification heat leak of 2150 BTU/hr.

This portion of the LOX system is actually flowing LOX during the vehicle fueling and replenish operations. The large 10-inch non-insulated line and other auxiliary lines were not considered for this analysis.

The total specification heat leak for the LOX V. J. system is

6-inch LOX V. J. lines - - - - -	12,200 BTU/hr
LUT Disconnect piping - - - - -	1,100 BTU/hr
8-inch LOX V. J. lines - - - - -	1,300 BTU/hr
10-inch LOX V. J. lines - - - - -	<u>2,150 BTU/hr</u>
Total =	16,750 BTU/hr
Total =	4,900 Watts

It will be assumed that the specification heat leak of 4900 watts represents an approximate total heat leak to the system defined. This includes all modes of heat transfer except pump work. Thus, $Q_o = 4900$ watts is the base heat leak to the system with a residual gas pressure of 10^{-4} torr within the vacuum spaces.

Next it will be determined what percentage of the 4900 watts is due to residual gas conduction within the vacuum spaces of the system. Again, this will be done by comparing the heat transfer at 10^{-4} torr to the heat transfer at 10^{-5} torr. To establish an arbitrary base, it will be assumed that there is zero heat leak due to residual gas conduction at 10^{-5} torr, and the difference in heat leak between 10^{-5} torr and 10^{-4} torr is the heat leak at 10^{-4} torr due to residual gas conduction.

The heat leak at various pressures will now be calculated. The surface area of the inner V. J. lines within the defined system is:

$$\begin{array}{rcl}
 1720 \text{ lineal feet of 6-inch V. J. lines} & = & 2984.1 \text{ ft}^2 \\
 21 \text{ lineal feet of 8-inch V. J. lines} & = & 47.4 \text{ ft}^2 \\
 26 \text{ lineal feet of 10-inch V. J. lines} & = & 72.2 \text{ ft}^2 \\
 \hline
 \text{Total Area} & = & 3103.7 \text{ ft}^2 \\
 & = & 2.8833 \times 10^6 \text{ cm}^2
 \end{array}$$

Average values for the apparent thermal conductivity for a multi-layer system will be taken from the experimental curves shown in figure 6-3. At 10^{-5} torr (\bar{k}) = $0.44 \mu \text{ watts/cm} - \text{K}$, and $Q = \frac{0.44 \mu \text{ watts}}{\text{cm} - \text{K}} \times \frac{210 \text{ K}}{2 \text{ cm}} \times 2.8833 \times 10^6 \text{ cm}^2 = 133 \text{ watts}$. At 10^{-4} torr, $\bar{k} = 0.55 \mu \text{ watts/cm} - \text{K}$, and $Q = 167 \text{ watts}$. The difference in heat leak calculated for the two pressure levels is 34 watts. Hence, it will be assumed that the heat leak due to residual gas conduction for the total LOX system at a pressure of 10^{-4} torr is 34 watts, and the heat leak due to all other modes of heat transfer is $4900 - 34 = 4866 \text{ watts}$. At 10^{-4} torr, the

percent of total heat leak due to residual gas conduction is

$$\frac{34}{4900} = .007 = 0.7\%.$$

The heat transfer due to residual gas conduction at pressures of 1, 10, 100, and 1000 microns was calculated in the same manner as for the liquid hydrogen system. Heat leak values of 138, 1199, 12887, and 26505 watts were calculated for the respective vacuum space pressures. This information is tabulated below, showing the ratio of Q/Q_o and the percent increase in system heat leak for the various vacuum levels.

Table 6-3

Liquid Oxygen System Heat Leak

vacuum space pressure (microns)	Q(watts) gas cond.	Q(watts) syst, total	Q(BTU/hr) syst, total	Q/Q_o	% increase in total syst. heat leaks
0.1	34	$Q_o = 4,900^*$	16,723	1	Zero
1	138		17,148	1.03	2.5%
10	1,199		20,700	1.24	24 %
100	12,887		60,590	3.62	286 %
1000	26,505		107,068	6.4	540 %

* Includes 34 watts due to gaseous conduction.

A plot showing the effect of vacuum space pressure on the total system heat leak is shown in figure 6-5. The above data indicates that pressures to one micron cause no significant increase in system heat leak. At pressures of 10 microns and above, the increase in system heat becomes appreciable.

6.4 Conclusions

It should be pointed out that the calculated values for heat leak should be considered as best estimates. Assuming that the actual heat

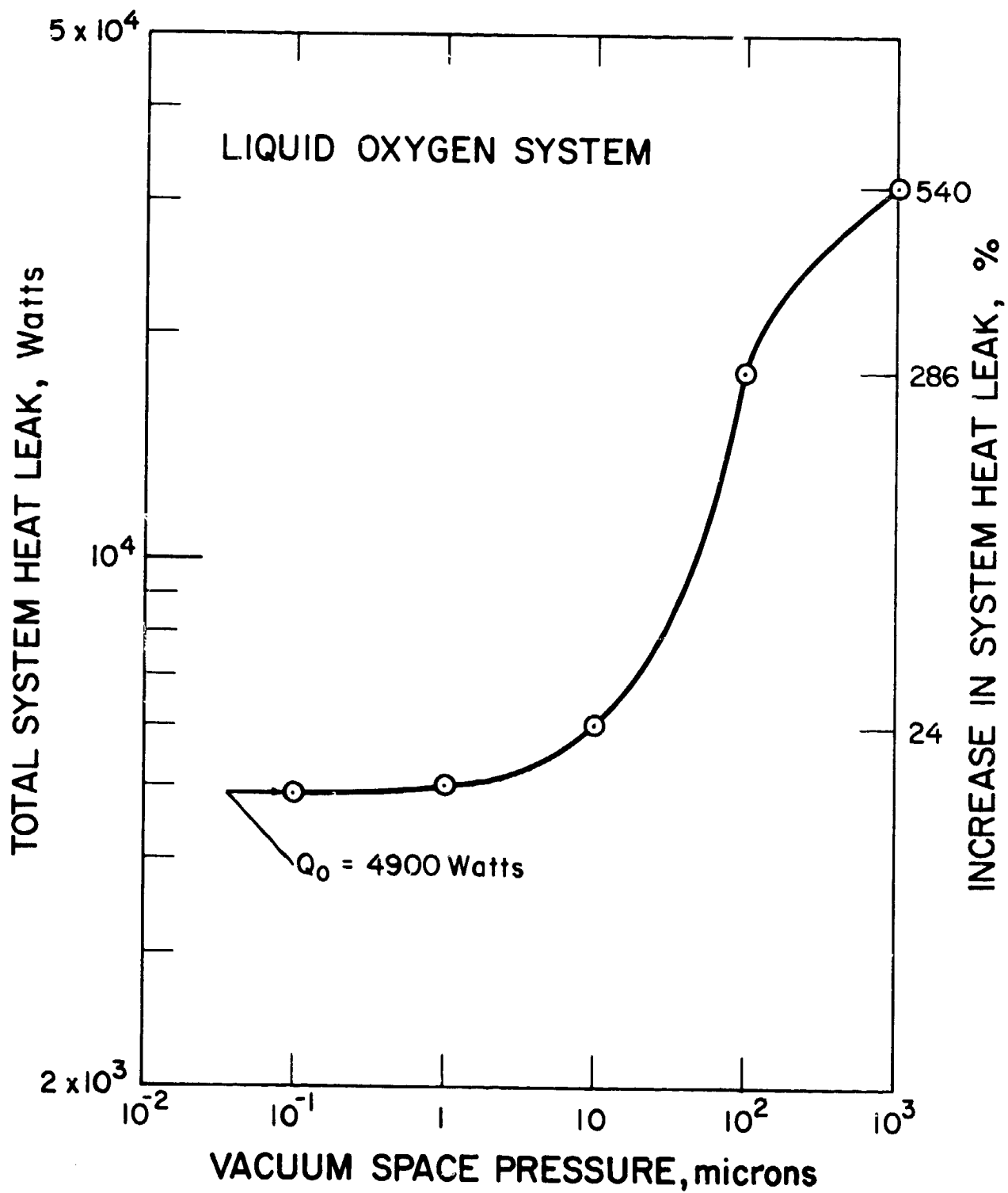


Figure 6-5 Liquid Oxygen System Heat Leak vs. Vacuum Space Pressure.

leak for the system is near the specification heat leak is an approximation. It is felt that this is a fair, good approximation; however, for this analysis, an accurate value for the system heat leak is not that important. We are merely using the system heat leak as a basis for comparative calculations. The experimental values for the apparent thermal conductivity given in figures 6-2 and 6-3 are undoubtedly lower than those achieved in a field installation such as the transfer lines at LC-39, where there are protrusions, inner line supports, insulation joints, and possibly compressive loads, all adversely affecting the performance of the multi-layer insulations. However, the difference in the apparent thermal conductivity due to increased interstitial pressure is the parameter of prime importance, and the relative values taken from the experimental data are very applicable in this analysis.

The data given in tables 6-2 and 6-3 are similar to what one would expect in looking at figures 6-2 and 6-3. Interstitial pressures to one micron have little effect on system heat leak. At 10 microns, the hydrogen system experiences a 10% increase in system heat leak, and the oxygen system experiences a 24% increase in system heat leak. Above 10 microns, the heat load rises very fast in both systems. The hydrogen system appears to be a little more tolerant of pressure in the 10-micron region.

This analysis shows that the heat leak due to gas conduction is a very important parameter. At a pressure of 10^{-4} torr, heat leak due to residual gas conduction is less than 1% of the total system heat leak. At pressures greater than 10 microns, the heat leak due to residual gas conduction rises very rapidly, and at pressures near 1 torr, the heat leak reaches values 4 to 6 times the total base system heat leak. Thus,

it is most important to keep the vacuum space pressure below 1 micron for minimal heat transfer. Although this analysis indicates that both of these systems would experience only a small percent increase in total system heat leak at 1 micron vacuum space pressure, the value of 10^{-4} torr (0.1 microns) has been used as the desired operating pressure level for this study.

7.0 Cryosorption Characteristics of Activated Charcoal and Molecular Sieve 5A

The ability of the sorbents inside the vacuum spaces of the transfer line sections at KSC to cryosorb the residual gases when cooled to cryogenic temperatures is most important in determining the acceptable pumpout level at ambient temperatures. These lines have a fixed radiant heat leak and a fixed heat leak due to solid conduction. The heat leak due to residual gas conduction can be extremely high, as pointed out in section 6, or almost negligible at pressures $\leq 10^{-4}$ torr. Hence, the cryosorption characteristics of the sorbents at their respective operating temperatures become the major factor in determining a maximum allowable pressure level at ambient temperatures.

Activated coconut charcoal and molecular sieve type 5A are the sorbents of most concern in this study, since they are the sorbents used in the transfer line vacuum spaces at KSC. These sorbents are very popular and have the following general sorption characteristics. Charcoal and molecular sieve have extremely large surface areas such as 1050 - 1100 m²/gram for A-C coconut charcoal [Juodbalkis, 1970] and 700 - 800 m²/gram for type A molecular sieve [Hersch, 1961]. The cryosorption capability of a sorbent is very dependent upon the type of gas to be sorbed and the temperature of the sorbent. In general, sorbents have a large sorption capacity for a gas near its normal boiling temperature, and this increases as the temperature decreases.

Because of the temperature dependence of the cryosorption capacity, it is most important that the sorbent have good thermal contact with the cold surface. A sorbent with a high bulk thermal conductivity is desirable in this respect, because the bulk of the sorbent cools faster. The sorption of most gases is reversible. Both sorbents have high capacity for water at ambient conditions. Molecular sieves, and probably charcoal too, have significantly reduced sorption capacity for other gases when the presorbed water exceeds 2 weight percent.

Nothing conclusive can be said about the comparative pumping rates of molecular sieves and charcoal for hydrogen gas at 20 K. Hemstreet, et al., [1964] investigated the pumping rates of these two sorbents. The molecular sieves had an apparent higher pumping rate but the charcoal was not properly shielded from thermal radiation during the tests, and the pumping speed is very dependent upon the temperature of the charcoal. For this evaluation, it will be assumed that coconut charcoal and molecular sieve 5A have similar pumping rates for hydrogen, oxygen, and nitrogen.

Another characteristic or phenomenon peculiar to molecular sieves and charcoal is a "conditioning effect," wherein the sorption capacity increases markedly after consecutive sorption-desorption cycles, and reproducible true equilibrium isotherms are obtained only after a number of these cycles. Stern [1964, 1967] observed the "conditioning effect" with both nitrogen and hydrogen on molecular sieves and coconut charcoal.

The effect is most pronounced at pressures below 10^{-4} torr and is independent of activation temperatures. Stern [1967] found that a reproducible sorption isotherm was obtained only after the fifth consecutive outgassing as shown in figure 7-1.

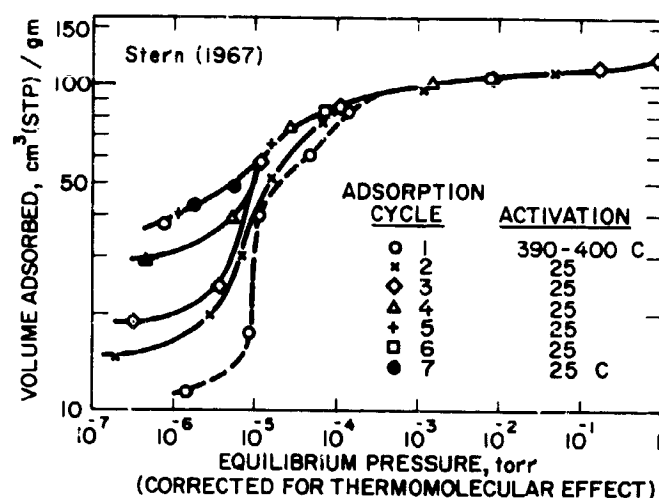


Figure 7-1 The Sorption of Nitrogen on Molecular Sieve 5A at 77.3K; Evidence of Conditioning Effect.

Gonzales [1955] also observed the "conditioning effect" during his work with nitrogen on charcoal. Stern suggests that the "conditioning effect" may be due to the gradual removal of some surface contaminant. The exact nature and cause of the "conditioning effect" is not understood at the present time. The decrease in sorption capacity at 10^{-4} torr pressure due to a non-conditioned sorbent is not significant, but it does become pronounced at pressures in the region of 10^{-6} torr.

The information most useful and readily available for evaluating and comparing sorbents is the equilibrium sorption isotherms for the gases of concern. These isotherms are obtained by admitting a given amount of gas to a chamber containing the activated sorbent at a known temperature and observing the equilibrium pressure after

equilibrium is attained. The experimental apparatus is usually ideal for cryosorption work in that the gas conductance is very good, and the freshly activated sorbent is located such that there is large surface area and very good thermal contact to the thin layers of sorbent. Most equilibrium sorption isotherms are obtained under near ideal conditions. The cryosorption conditions inside a transfer line vacuum space are quite different, especially when multi-layer is present inside the vacuum space. The flow conductance becomes very poor, and the sorbent is not ideally distributed, but hopefully there is very good thermal contact between the inner line and the sorbent.

The equilibrium sorption isotherms provide the sorption capacity of the sorbents for the various gases at various pressures and temperatures, and the work of several investigators is in fair agreement. The additional time required to achieve an insulating vacuum because of poor conductance is unknown and would probably have to be determined empirically. The equilibrium sorption isotherms will be used for this study since they represent the most useful data available for cryosorption evaluation, bearing in mind that they are obtained under ideal conditions.

7.1 Charcoal Cryosorption of Gases

Activated coconut charcoal is the sorbent used inside the vacuum spaces of the 10-inch LH_2 transfer lines of LC-39B. Coconut charcoal is a very good cryosorbent, but it has the disadvantage of being non-LOX compatible [Martindale, 1970]. Charcoal has a relatively high thermal conductivity and is not as expensive as some of the other sorbents. Charcoal has comparatively high sorption capacity for most gases at low partial pressures and temperatures near their normal boiling temperatures. The gases of most concern in a transfer line cryosorption application are nitrogen, oxygen, argon, neon, hydrogen,

and helium. The first four are constituents of air, and hydrogen is the principal gas due to metal outgassing as pointed out in section 4. Helium is a minor constituent of air but is still important because it is used most often for leak detection of vacuum spaces, and it is possible to leave small concentrations of helium in a vacuum space, especially if the space contains sorbents and glass paper between the multi-layer insulation. Hydrogen, helium, and neon are the most important of these gases because at 90 K they are very weakly sorbed by charcoal and molecular sieves. At 20 K they are more easily sorbed and are the only noncondensibles remaining (a noncondensable gas being defined as one that does not condense to the solid phase with a vapor pressure less than 10^{-11} torr). The pure equilibrium sorption isotherms of the above mentioned gases on activated charcoal are shown in figure 7-2. The sorption isotherms were determined by various investigators. The sorption capacity in the figure is given in cm^3 [STP] (273 K & 760 torr) per gram of sorbent, and the abscissa is the equilibrium pressure of the particular gas on the sorbent.

Probably the most important aspect of these sorption isotherms is the sorption capacities of hydrogen and neon at 20 and 77 K. Activated charcoal at 20 K has a very high sorption capacity for hydrogen, and it is fairly constant over the pressure range from 10^{-6} to 10 torr. However, at the lower pressures, the sorption capacity decreases quickly with a relatively slight increase in temperature. For instance, in the 10^{-4} torr pressure region (where a good insulating vacuum begins), there is an order of magnitude difference in the sorption capacity between 20 K and 40 K, and there is over four orders of magnitude difference in the sorption capacity between 20 and 80 K. Because of this, it is most important to have very good thermal contact between

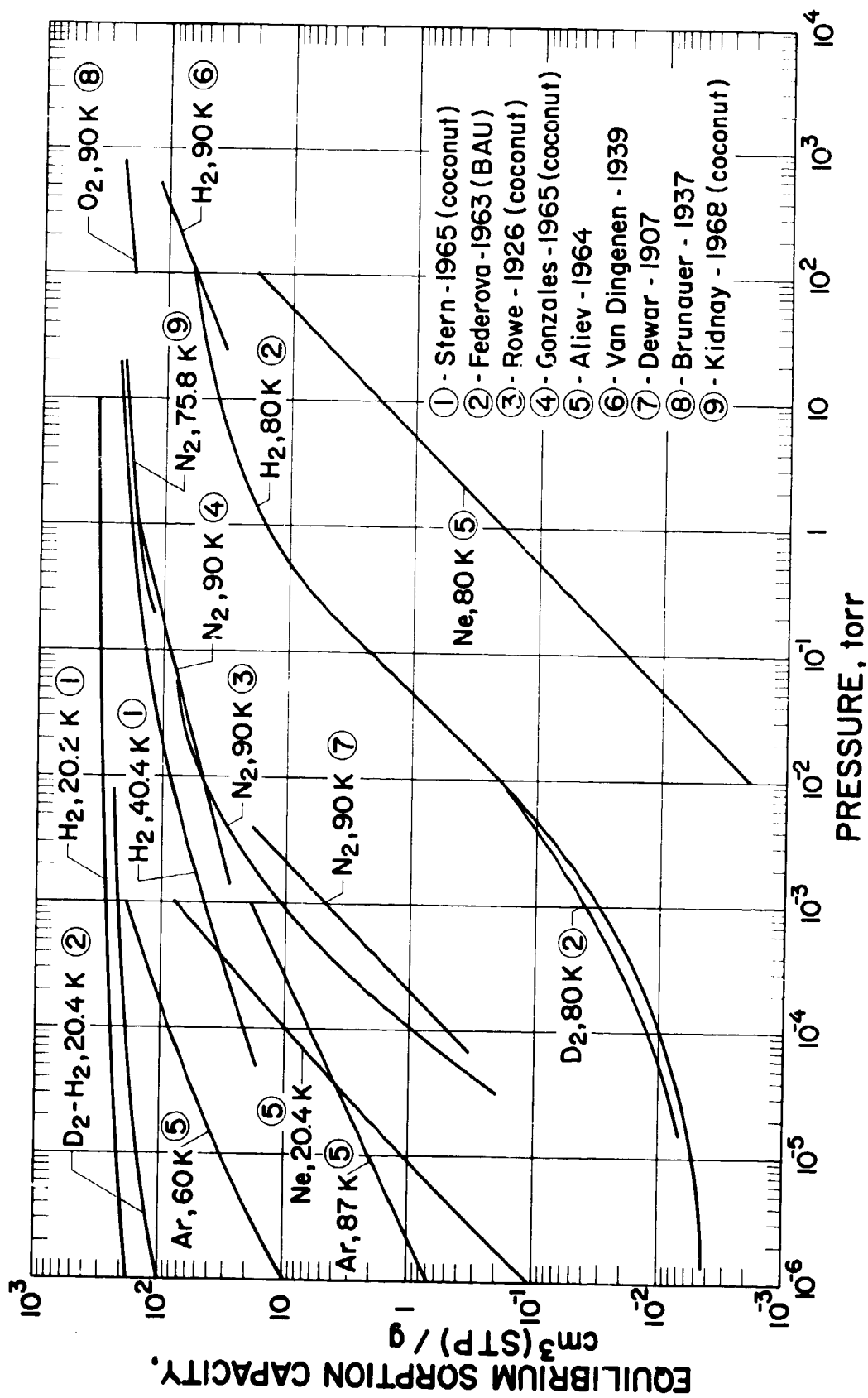


Figure 7-2 Equilibrium Sorption Isotherms for Various Gases on Activated Charcoal at 20 - 90 K.

the charcoal sorbent and the cold surface in a hydrogen transfer line or dewar application. This is also why a relatively high thermal conductivity sorbent is desirable. It is important to cool the bulk of the sorbent as soon as possible.

Neon also has the same type of temperature dependence as hydrogen. For instance, at 10^{-3} torr there is a difference in sorption capacity of five orders of magnitude between 20 and 80 K, and the sorption capacity at 90 K would be even less. The sorption of neon is very important, as will be shown later, because although neon is a minor constituent of atmospheric air, it becomes very important and predominates in the cryosorption of air, since neon strongly governs the ultimate attainable vacuum at 90 K.

The sorption capacity of charcoal for nitrogen, oxygen, and argon is usually very similar. There is very little data available on the sorption capacity of oxygen at low temperatures and pressures. The sorption capacity of oxygen at higher pressures of 1 to 760 torr is slightly higher than that of nitrogen. For this study, it will be assumed that the sorption capacity of oxygen is the same as nitrogen at 90 K and low pressures (10^{-5} to 10^{-2} torr). The sorption capacity of charcoal at 90 K for nitrogen is fair in the 10^{-4} torr pressure region, and at 20 K, nitrogen is readily condensed to a solid with a vapor pressure of $\approx 10^{-11}$ torr. The sorption capacity of charcoal at 87 K for argon is also good, and at 20 K, it is condensed to solid with a very low vapor pressure ($\approx 10^{-11}$ torr). Although no sorption data for helium on charcoal were found, the sorption isotherms would probably be very similar to those for molecular sieves, as shown in figure 7-3. Helium and neon are both very weakly sorbed near 90 K at pressures from 10^{-5} to 1 torr.

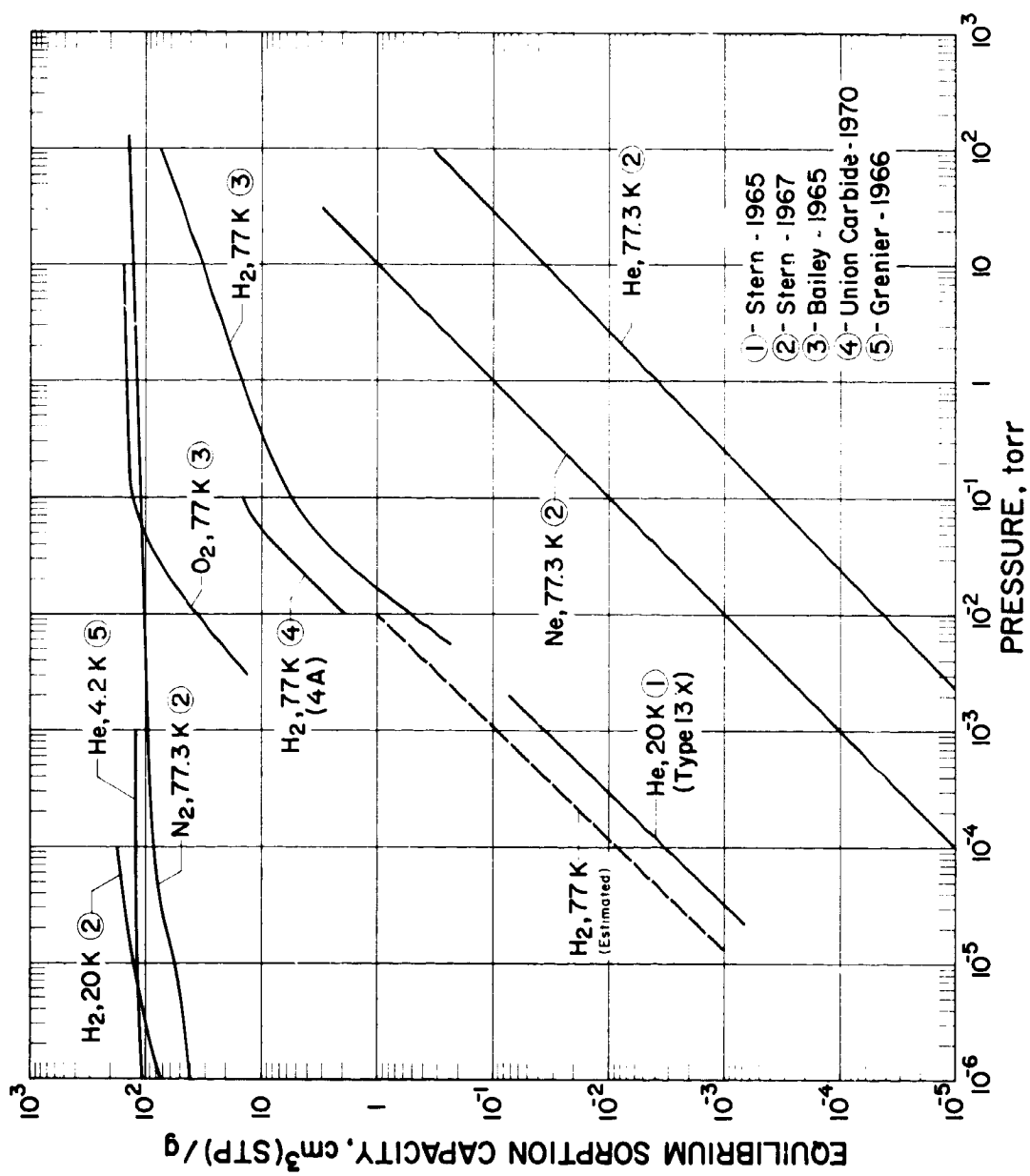


Figure 7-3 Equilibrium Sorption Isotherms for Various Gases on Molecular Sieve 5A at 4 - 77 K.

Another important sorption characteristic of charcoal is that the sorption of oxygen is somewhat irreversible. Lowry [1920] investigated the sorption and desorption characteristics of oxygen on charcoal and found that only approximately 50% of the oxygen sorbed at 25°C could be desorbed by evacuating at room temperature, and only traces of oxygen could be desorbed when the temperature was increased to 184°C. Increasing the temperature to 900 and 1000°C caused the charcoal to desorb carbon monoxide and carbon dioxide. It was suggested that the oxygen atoms are chemically bound to the surface carbon atoms, forming carbon dioxide and carbon monoxide which can be removed only by heating to 900 - 1000°C. Very little work has been done on this anomaly, and the effect of this incomplete desorption of oxygen will not be considered in this study.

7.2 Molecular Sieve Cryosorption of Gases

Molecular sieve is a common name given to a group of synthetically produced crystalline metal alumina-silicates. Type 5A molecular sieve is an alkali metal alumina-silicate and has the calcium form of type A crystal structure. Type 5A molecular sieve was the sorbent used in the major portion of the transfer line vacuum spaces at LC 39A and B. All the molecular sieve sorption data in this report, unless otherwise noted, is for type 5A. Molecular sieves have a distinct advantage over most other sorbents in that the pores of any particular type are precisely uniform in size. For instance, type 5A will sorb molecules with critical diameters up to 5 angstroms, and 13X will sorb molecules with critical diameters up to 10 angstroms. Using this property, it is possible to selectively filter out certain molecules by using the appropriate sieve.

Sorption isotherms for molecular sieves at low temperatures and pressures are not abundant in the literature. However, recent interest in cryosorption has prompted investigation into the sorption characteristics of molecular sieves at low temperatures and pressures. Sorption isotherms for various gases on molecular sieve near 20 and 77 K are shown in figure 7-3. The isotherms presented are for the 1/16 and 1/8 in. diameter pellets which contain 20 weight percent of inert clay binder.

The sorption capacity of molecular sieves for hydrogen at 20 K and pressures $\leq 10^{-4}$ torr is slightly less than that of coconut charcoal. The sorption capacity of molecular sieves at 77 K for hydrogen is similar to charcoal. Type 5A molecular sieves have very good sorption capacity for hydrogen at 20 K and nitrogen, oxygen, and argon at 77 K. As with charcoal, helium and neon are very weakly sorbed at 77 K. At 20 K, the sorption capacity for helium is considerably higher than at 77 K, but it is still weakly sorbed at low pressures, compared to most other gases. No sorption isotherms for neon on molecular sieve at 20 K have been found, but it is a fair assumption that the sorption capacity is approximately the same as that of charcoal. In general, it appears that the sorption characteristics for the gases to be sorbed in a transfer line application are approximately the same for charcoal and molecular sieve 5A. However, the water sorption and desorption characteristics of the two sorbents are not the same.

7.3 Water Sorption Characteristics

There is a considerable difference between the water sorption characteristics of charcoal and molecular sieves, especially at low partial pressures of water. The water sorption characteristics of these sorbents is very important because of the high humidity normally present at KSC. The KSC Weather Bureau [1970] records an average

year round relative humidity of 80%, varying only 6 - 8% over the year. At 80% relative humidity and 25°C, the water partial pressure is ≈ 19 torr. Conceivably, there could be times when the transfer line vacuum space is exposed to the atmosphere for 1 or 2 days during repair. Also, large leaks to the vacuum spaces may occur from time to time. Because of this, there exists a distinct possibility of saturating the sorbent with water, which can result in a significant decrease in sorbent capacity. The water sorption and desorption characteristics of charcoal and molecular sieves will be compared to determine the effect of atmospheric exposure on the sorbents.

7.3.1 Activated Charcoal

Some typical sorption isotherms for water vapor on charcoal are shown in figure 7-4. All of the sorption isotherms presented by Allmand [1924] and Coolidge [1927] for water vapor on various types of charcoal at different temperatures have the same typical shape as those shown in figure 7-4. Most of the charcoals investigated by Coolidge and Allmand had a maximum sorption capacity of approximately 400 milligrams of water per gram of charcoal near ambient temperatures (40 weight percent). Both investigators observed that sugar charcoal had only about 50% the capacity of coconut and other charcoals. The maximum sorption capacity appears to be a function of the type of charcoal, the ash and silica impurities within the charcoal, and the charcoal extraction procedures. All the isotherms have a very steep section below which the sorption capacity levels off to a very low value. The portion of the curve below 4 torr is most important for this study. There appears to be no ambient temperature water sorption data available for charcoal at pressures below one torr. When the water sorption isotherms are presented on a plot as shown in figure 7-4, the curves

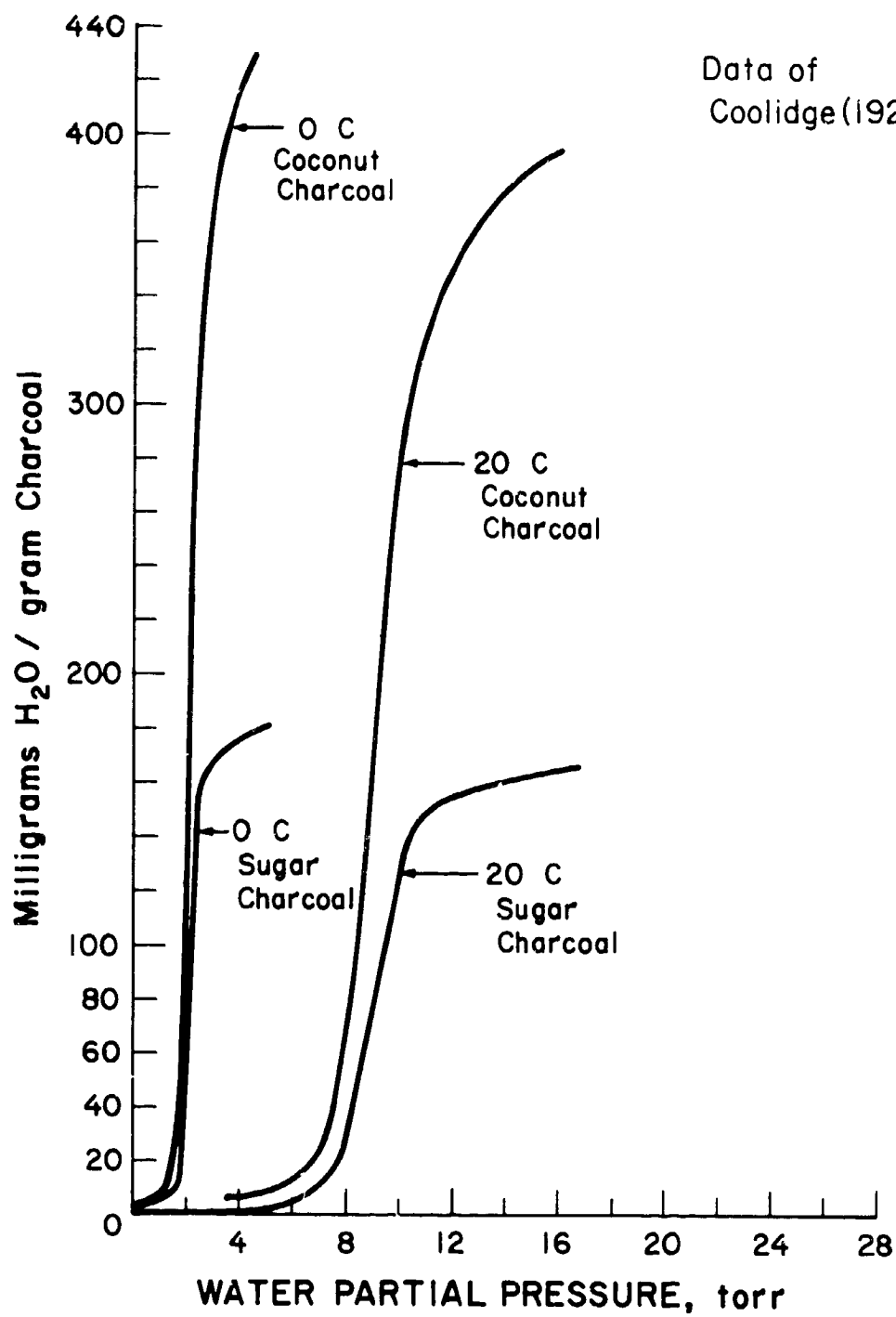


Figure 7-4 Equilibrium Sorption Isotherms for Water on Activated Charcoal.

move toward the right with increasing temperature. At higher temperatures, the ultimate capacity decreases, and the lower portion of the curve spans a larger partial pressure. The water desorption isotherms are very similar in shape to the sorption isotherms, and usually a slight hysteresis loop is present. From the water sorption and desorption work by Allmand and Coolidge, it is quite apparent that water is readily sorbed and desorbed near ambient temperatures (0 - 25C), although no specific sorption and desorption rates are given.

With this basic knowledge of charcoal water sorption characteristics, it is conceivable that if a vacuum space containing charcoal were exposed for 1 - 2 days to atmospheric air of high humidity as found at KSC, the charcoal could sorb as much as 400 milligrams H_2O /gram charcoal or approximately 40 weight percent of water. However, the sorption curves indicate that when the partial pressure of water is reduced to ≈ 4 torr at ambient temperature, the sorption capacity decreases drastically. Assuming the above vacuum space was repaired, sealed, and pumped to $\approx 10 - 50$ microns (normal procedure), most of the water should desorb (there is a slight hysteresis effect) and leave the charcoal with a very low weight percent of water. Thus, charcoal has the ability to sorb large quantities of water vapor in the event of exposure to high humidity atmospheric air, but the water is also easily desorbed from the charcoal by normal pumping procedures at ambient temperatures. Desorbing the water from a vacuum space containing multi-layer could require a long pumping period due to poor conductance, but no elevated temperatures are required for desorption.

There is another aspect of water sorption on charcoal that should be mentioned. Several investigators have noted a hysteresis effect when desorbing water vapor from charcoal. Allmand [1929] investigated this thoroughly and found that the water nonrecoverable at 25°C ranged

from 1.6 to ≈ 10 milligrams water/gram of charcoal, for charcoal which had experienced water vapor pressure up to 23.8 torr. Bachman [1917] and Rakovsky [1917] have also reported this hysteresis effect with water. Evidently, the hysteresis effect is not completely understood, but there is considerable experimental data to substantiate its existence. At any rate, the highest value of 10 milligrams of water retention/gram of charcoal amounts to one weight percent. Pumping to $\approx 10 - 50$ microns, as in a vacuum space, should further reduce this, and if not, the work on molecular sieves tends to indicate that one weight percent presorbed water does not seriously reduce the sorption capacity for nitrogen gas, and one could expect similar characteristics for charcoal.

7.3.2 Molecular Sieve 5A

The water sorption and desorption characteristics of most molecular sieves are considerably different from those of charcoal. Molecular sieves have tremendous capacity for water at very low partial pressures, and this is one of their major attributes. However, because the water is so strongly sorbed, it is also not easily desorbed at ambient temperatures. Figure 7-5 is a water sorption isotherm plot for molecular sieve 5A [Union Carbide-a, 1970].

If the same conditions are assumed as with charcoal, namely the possibility of the sieves being exposed to high humidity atmospheric air for a period of 1 - 2 days during repair or due to a large leak occurring in the outer line of the vacuum space, the amount of water that could be sorbed may be determined from the sorption isotherms shown in figure 7-5.

It will be assumed that the water partial pressure of the atmospheric air at KSC is 19 torr. The sorption data indicates that the molecular sieves have a water capacity of 20 weight percent at a water

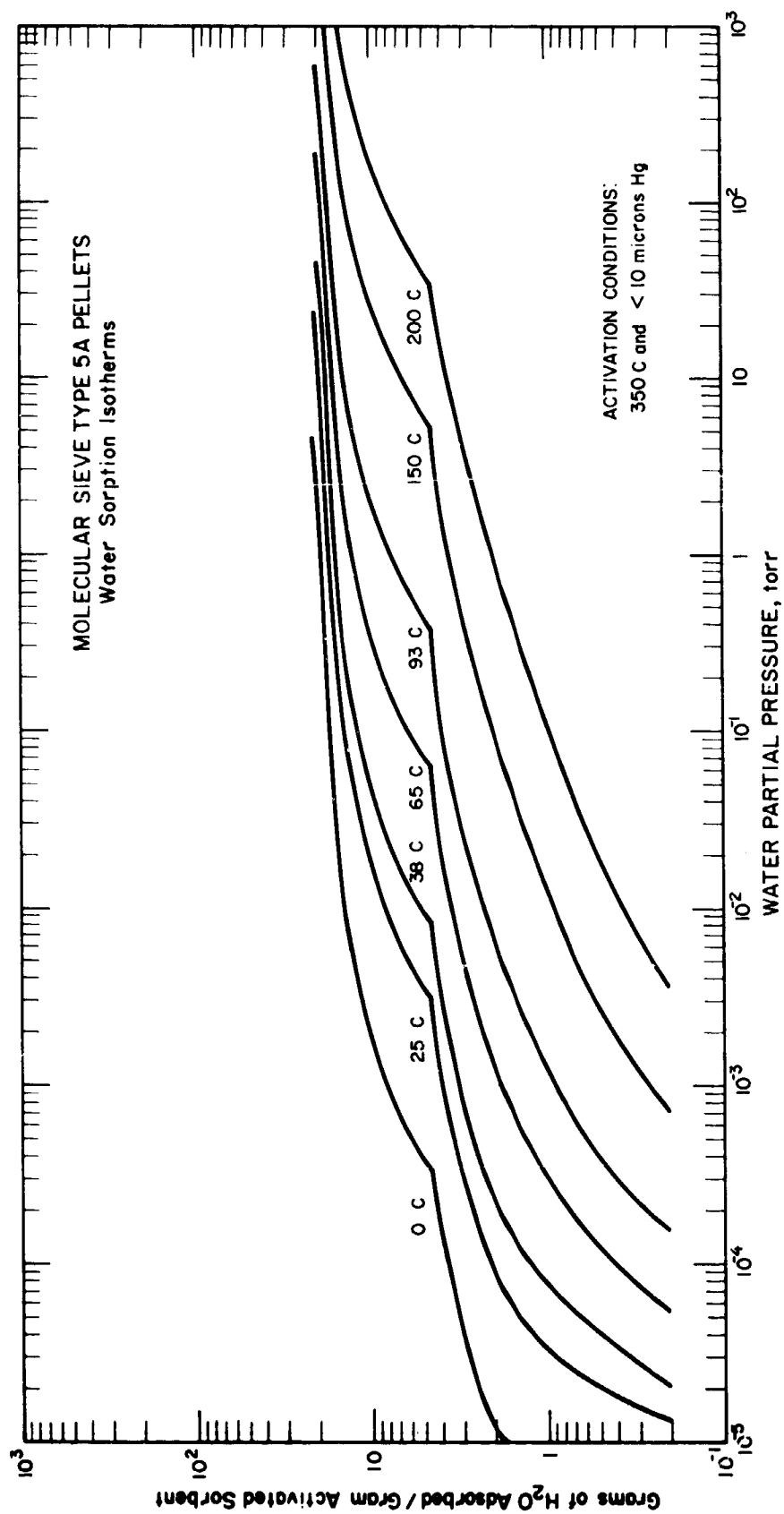


Figure 7-5 Equilibrium Sorption Isotherms for Water on
Molecular Sieve 5A.

partial pressure of 19 torr. If it is further assumed that the repair was performed, the vacuum space sealed and pumped to ≈ 10 microns pressure, the water still present in the sieves can be determined from the 25°C isotherm at 10 microns pressure. It would be further assumed that the water partial pressure is equal to the total pressure after being pumped (for a period of time) to 10 microns. The water sorption capacity of the sieves at 25°C and 10 microns is still 8.2 weight percent. At 8.2 weight percent water, the sorption capacity of molecular sieves at 77 K for nitrogen is drastically reduced as will be shown later. If the vacuum space were pumped to 1 micron pressure, which would be difficult if the sieves contained water, the sieves would still hold 4 weight percent water.

So it becomes readily apparent that conventional pumping at ambient temperatures to pressures in the range of 10 to 50 microns is going to leave the sieves with around 8 weight percent water, which drastically reduces their sorption capacity. Heating the sieves to 150°C and pumping to 10 microns pressure reduces the water sorption capacity to one weight percent, and heating to 200°C reduces the water sorption capacity to ≈ 0.4 weight percent. The recommended activation conditions for molecular sieves [Union Carbide-a, 1970] are 350°C and < 10 microns pressure.

Hence, there is a significant difference in the water desorption characteristics between charcoal and molecular sieves. If charcoal becomes loaded with water due to exposure to the atmosphere, it can be pumped in the usual manner (to 10 or 50 microns pressure) and the water will desorb to a very low weight percent. It may require 2 - 3 days to pump to 10 microns pressure, but the charcoal will be activated and ready to perform with near rated sorption capacity for hydrogen, nitrogen, and the other important gases.

If molecular sieves become loaded with water due to atmospheric exposure, and are pumped in the usual manner to 10 - 50 microns, the sieves may still contain as much as 8 weight percent water, which drastically reduces their rated sorption capacity for the above gases. Pumping water from molecular sieves thru multi-layer insulation to $\approx 10^{-5}$ torr is extremely difficult. It is apparent that a vacuum-bakeout at a minimum of 150 - 200°C is necessary to effectively desorb the water from molecular sieves.

One might argue that the molecular sieves would only be exposed to atmospheric air for a maximum period of 8 hours while repairs were performed, and this short exposure time is not sufficient for the sieves to sorb a significant amount of moisture. Bailey [1965] determined the rate of sorption of water vapor from the laboratory atmosphere on a freshly activated 20 gram sample of molecular sieve 5A. The sample was baked overnight at a temperature of 315°C, cooled, and exposed in an open cylindrical vessel of 6.6 cm diameter x 2.5 cm high. The laboratory temperature was 24°C and the relative humidity was 57% (12.7 torr water partial pressure). The rate of free sorption is shown in figure 7-6.

The term "free sorption" is used because a small amount of gas would be sorbed along with the water. However, since oxygen, nitrogen, and argon are weakly sorbed at ambient conditions, and carbon dioxide and water are strongly sorbed, most of the weight increase would be due to water, since there is only 0.03 percent, by volume, carbon dioxide in air. After 8 hours of exposure, the sieve had sorbed approximately 6 weight percent water. After 24 hours of exposure, the sieve had sorbed approximately 9 weight percent water, and after 72 hours, the sieve had sorbed 22 weight percent. Shortly after 72 hours, the sieve attained maximum sorption capacity of ≈ 25 weight percent.

The equilibrium sorption capacity of molecular sieve 5A for water, from figure 7-5, at 10 torr partial pressure is 22 weight percent. At KSC, the average relative humidity is $\approx 80\%$, corresponding to a water partial pressure of 19 torr, which is 50% higher than the water partial pressure of Bailey's laboratory. From the above data, one can easily argue that molecular sieves exposed to the open atmosphere at KSC for a work period of 8 hours could easily sorb 10 weight percent water.

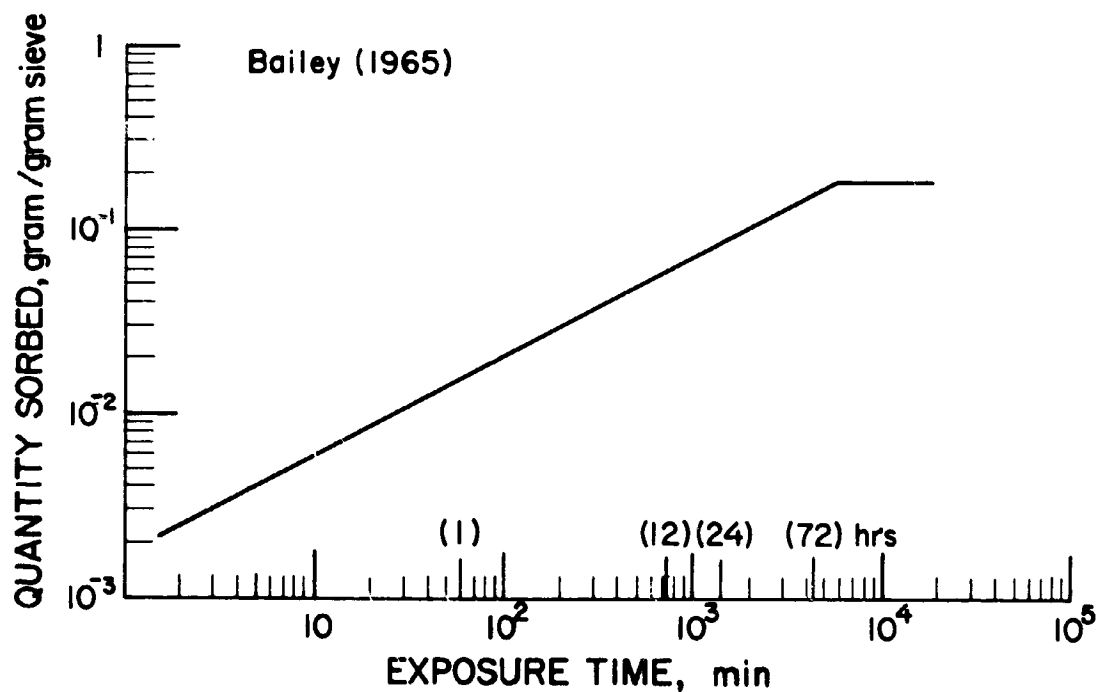


Figure 7-6 Free Sorption Rate of Molecular Sieve 5A Exposed to Laboratory Atmosphere (24 C and 57% relative humidity).

7.4 Effect of Presorbed Water on the Sorption Capacity of Molecular Sieves

Since molecular sieves have such a strong attraction for water, even at low partial pressures, the possibility of presorbing water prior to the main cryosorption application is very high. Water could be easily presorbed by exposing the sieve to high humidity atmospheric air during assembly or repair to a vacuum insulated vessel or transfer line. Because of the strong attraction of molecular sieves for water, several workers have investigated the effect of presorbed water on the equilibrium sorption capacities of molecular sieves for other gases.

Stern [1967] determined the effect of presorbed water on the sorption characteristics of nitrogen gas at 77 K. Stern found that with 2 weight percent presorbed water, a reduction in the N_2 sorption capacity appeared. At 7 weight percent water, the decrease in sorption capacity for N_2 was an order of magnitude lower than that of the activated molecular sieve. At 10 weight percent, the N_2 sorption capacity was seriously decreased, as shown in figure 7-7.

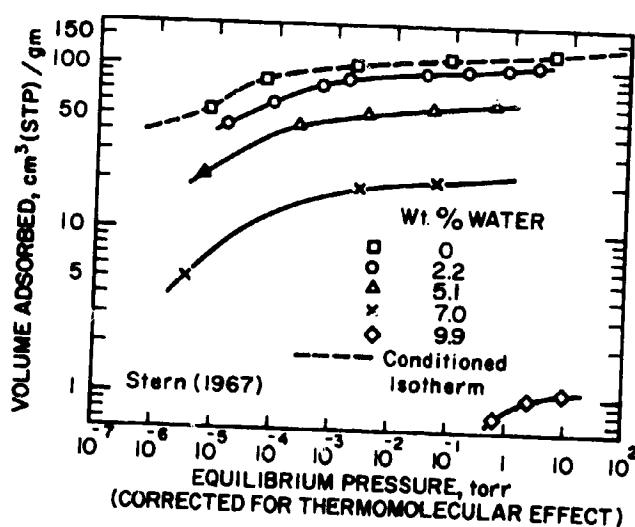


Figure 7-7 Effect of Presorbed Water on the Sorption of Nitrogen on Molecular Sieve 5A at 77.3 K.

The "conditioned" isotherm for nitrogen obtained in the absence of water is shown with broken lines. Stern concluded that above 7 weight percent presorbed water, the sorption capacity of molecular sieve 5A at 77 K for nitrogen is almost completely destroyed. Stern suggests that, "this behavior is due to the migration of hydrated cations which results in blocking of the apertures of the main cavities of the molecular sieve." Hemstreet [1964] investigated the effect of presorbed water on hydrogen cryosorption pumping with molecular sieve 5A. Hemstreet and his co-workers were mainly concerned with the cryosorption rate (pumping speed) of the molecular sieve and not the equilibrium sorption capacity. Hemstreet only conducted a few tests with presorbed water, but he concluded that presorbed water can drastically reduce the cryosorption pumping speed of hydrogen on molecular sieve 5A.

From the preceding work by Stern and Hemstreet, concerning the effect of presorbed water on the sorption capacity of molecular sieves for nitrogen and hydrogen, it is reasonable to assume that a similar reduction in sorption capacity would also occur for other gases of interest such as neon, oxygen, argon, and helium.

Charcoal too, is undoubtedly susceptible to reduced sorption capacity due to exposure and presorbed water, but the big difference with charcoal is that most of the water may be desorbed by merely pumping at ambient temperatures, whereas with molecular sieves, this is not possible. The point of most importance in this section is that molecular sieves are very susceptible to water sorption if exposed to atmospheric conditions such as the high humidity environment at KSC. The water cannot be desorbed by pumping at ambient temperatures, and the sieves can sustain a drastic reduction in sorption capacity which could decrease the sorption capacity orders of magnitude

below the design performance rating. Because it is extremely difficult to desorb water from molecular sieves at ambient temperature, it is recommended that the vacuum spaces containing molecular sieves that are suspected* of containing more than 2 weight percent presorbed water should be given a vacuum-bakeout at a minimum temperature of 150 - 200°C to restore the cryosorption capacity of the sieves to their normal activated capacity. The recommended activation conditions are 350°C and < 10 microns pressure.

Reliably determining the amount of water presorbed by molecular sieves is not straightforward. The sorption isotherms in figure 7-5 indicate that at 25°C, the sieves should have a water partial pressure of 10^{-4} torr when they contain 2 weight percent water and equilibrium conditions exist. A fairly sensitive mass spectrometer or moisture analyzer would be required to determine the partial pressure of water in this pressure region. Also, it could take days for equilibrium to establish after repairing and pumping the vacuum space. It may be that carefully examining the pressure and leakage history of the vacuum space, and calculating the moisture sorbed due to leakage, would be as good a method as trying to analyze the moisture present inside the vacuum space. Simple laboratory tests could be made at 80% relative humidity to determine the water sorption rate of molecular sieves at the KSC environment.

Recommending a vacuum-bakeout is straight forward but is not very easily accomplished. It should be remembered that the molecular sieves have to be heated to the recommended temperature, and that the sieves have very good thermal contact with the inner line, with multi-layer insulation between them and the outer wall. Heating the

* As determined by vacuum maintenance history, i. e., vacuum level, suspected leakage, repair record, etc.

sieves to the recommended temperature, using an exterior heat source, could take a long time, even though 10 microns pressure is well above a good insulating vacuum. It would be preferable to heat the inner line and simply pump out the vacuum space of the section containing water; circulating hot gas through the shortest possible length of line is one possibility. At any rate, an effective vacuum-bakeout is not easily accomplished and will require considerable effort if the treatment is to be effective.

There is considerable discussion in this report about high temperature vacuum-bakeouts, vacuum-bakeouts at 250 - 300°C to outgas hydrogen from metal, and vacuum bakeouts at 350°C to desorb water from molecular sieves. It should be clearly pointed out that the upper temperature limit of Mylar is 138°C (280°F) [Norton Co., 1970], which is considerably lower than the bakeout temperatures above. This is a distinct disadvantage in using aluminized Mylar inside a vacuum space, and it should certainly not be used with molecular sieves in a high humidity environment where there is a good possibility of the sieves becoming saturated with water. The specifications and information available indicate that all the vacuum spaces at KSC containing aluminized Mylar also contain coconut charcoal for a sorbent, so any water within these vacuum spaces may be easily desorbed at ambient temperatures.

Another disadvantage of Mylar is that it is non-LOX-compatible and should not be used inside the vacuum space of a LOX transfer line. Riehl [1970] of NASA points out that Mylar is still considered non-LOX compatible, and since the Apollo 13 incident, they are taking a closer look at aluminum foil. They currently consider aluminum foil and glass fiber cloth LOX compatible and acceptable for use as multi-layer insulation in the vacuum spaces of LOX transfer lines with

operating pressures less than 50 psig. Riehl adds, however, that they are "not comfortable" about using aluminum foil in systems with operating pressures greater than 1000 psig.

7.5 Effect of Presorbed Dry Air on the Cryosorption of Hydrogen by Molecular Sieves at 20 K

Hemstreet [1964] investigated the effect of presorbed dry air on the cryosorption pumping rate of hydrogen by molecular sieve 5A at 20 K. For this experiment, the dry air was sorbed at temperatures down to 76 K while the sorbent was cooled with liquid nitrogen. The residual gas was then pumped out with diffusion pumps; the nitrogen coolant was replaced with hydrogen, and the hydrogen cryosorption experiment started. The cryosorption pumping rate of hydrogen on molecular sieve 5A, with varying amounts of presorbed dry air, was compared to the pumping rate of the uncontaminated sorbent, as shown in figure 7-8. During this experiment, a steady hydrogen leak rate of one micron-liter/minute was admitted to the cryosorption apparatus while the pressure was recorded. The lower curve represents the hydrogen pressure vs. time for the uncontaminated molecular sieve. The upper curves indicate the effect of varying amounts of presorbed dry air on the pumping rate of the sorbent. Hemstreet concluded that, as long as the volume of presorbed air does not exceed $30 \text{ cm}^3 \text{ [STP]/gm}$ of sorbent and binder, the cryosorption rate is essentially the same as with freshly activated sieve. Above $30 \text{ cm}^3 \text{ [STP]/gm}$, there was a significant decrease in the cryosorption pumping performance of the sorbent. This data is obviously a function of the apparatus, but it clearly demonstrates the effect of presorbed dry air on the pumping rate of molecular sieve 5A for hydrogen at 20 K.

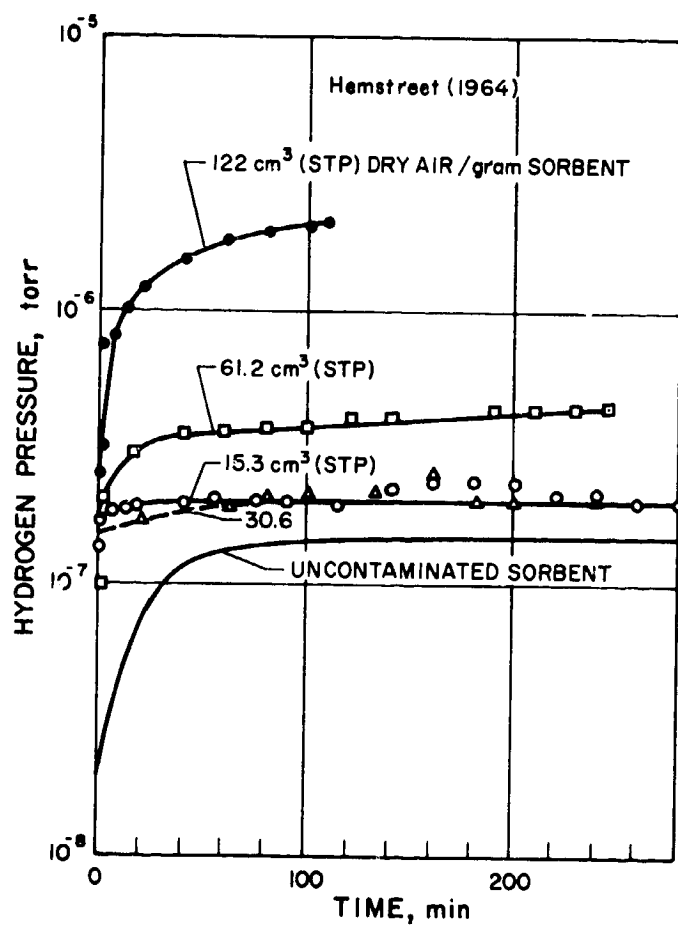


Figure 7-8 Effect of Presorbed Air on Hydrogen Cryosorption Pumping by Molecular Sieve 5A.

The effect of presorbed air on molecular sieves is very important in this study of the cryosorption characteristics of the transfer line vacuum spaces at KSC. It is conceivable that a transfer line vacuum space at 4000 microns pressure could have 80% air due to a small leak and 20% hydrogen due to metal outgassing. As the transfer line and sorbent cools toward 20 K, the N_2 , O_2 , and Ar constituents of air will be very strongly sorbed in the region 70 - 90 K, while the hydrogen is still quite weakly sorbed. As the sorbent further cools to 20 K, the hydrogen is then strongly sorbed, provided the pore openings are not blocked due to air constituents previously sorbed at higher temperatures. Hemstreet's work indicates that there is no blocking of the sorbent pores as is apparently the case with ≈ 10 weight percent presorbed water on molecular sieves, and that below a level of 30 cm^3 [STP]/gm of presorbed air, the pumping rate of hydrogen at 20 K is not seriously affected. The transfer line vacuum spaces at KSC have at least 1000 grams of sorbent in each vacuum space, and at 4000 microns pressure, each vacuum space has no more than 1225 cm^3 [STP] of gas. If it is assumed that most of the gas is air, a calculation reveals that $30 \text{ cm}^3/\text{gram} \times 1000 \text{ grams} = 30,000 \text{ cm}^3$ [STP] of air may be presorbed with no serious reduction to the hydrogen sorption rate at 20 K. Since there is only 1225 cm^3 [STP] total gas to be sorbed in each vacuum space at 4000 microns pressure, it appears that the cryosorption rate of hydrogen at 20 K would be affected very little by the air presorbed at higher temperatures. The final equilibrium sorption capacity of the sieve for hydrogen would be decreased due to the presorbed air, but a simple ratio calculation of sorption capacities indicates this to be less than one percent. Although no similar work has been done with charcoal, it will be assumed that molecular sieve 5A and coconut charcoal have similar cryosorption characteristics, when considering the effect of presorbed dry air on hydrogen sorption at 20 K.

7.6 Comparative Cryosorption Characteristics of Charcoal and Molecular Sieve 5A

A table showing the comparative characteristics of importance in a cryosorption application for charcoal and molecular sieves is given below.

Table 7-1
Comparative Cryosorption Characteristics of Coconut Charcoal
and Molecular Sieve 5A

Sorbent Characteristics	Coconut Charcoal	Molecular Sieve 5A
Water sorption at low partial pressures (\leq one torr and ambient temperatures)	Very poor	Excellent
Water desorption at ambient temperatures	Very good (slight hysteresis)	Poor (elevated temp. required)
LOX compatibility	Considered not acceptable	Acceptable
Thermal conductivity (Bulk)	Good	Poor
Price (1970) for 50#	\$48.00	\$65.50
Pore diameter	$\approx 17 \text{ \AA}$	5 \AA
Oxygen desorption	Poor	Excellent
Sorption capacity for H_2 at 20 K and 10^{-4} torr	Excellent	Very good
Sorption capacity for N_2 , O_2 , Ar at 90 K and 10^{-4} torr	Very good	Very good
Maximum temperature	$\approx 1000^\circ\text{C}$	600°C

Most of the parameters are self-explanatory or have been explained in detail in the text. Bulk thermal conductivity values for both sorbents have been unobtainable. Many investigators refer to the better thermal conductivity of charcoal but give no values at evacuated conditions. A maximum temperature of 600°C for molecular sieves is given by Hersh [1961], but he cautions that the sieves will only survive a few regeneration cycles at this temperature. The maximum temperature of coconut charcoal is around 1000°C [Lowry, 1920].

8.0 Sorbent Performance Study

8.1 Cryosorption of Air at 20 and 90 K

In evaluating a transfer line vacuum space subject to atmospheric leakage, the cryosorption of the various constituents of air and the moisture present in air become important. Table 8-1 shows the composition of dry atmospheric air on a volume and weight percent basis.

Table 8-1

Normal Composition of Clean, Dry, Atmospheric Air Near Sea Level
[U. S. Std. Atmosphere, 1962]

Constituent	Volume Percent	Weight percent	Molecular weight*
Nitrogen	78.084	75.521	28.0134
Oxygen	20.9476	23.142	31.9988
Argon	0.934	1.288	39.948
Carbon Dioxide †	0.0314	0.048	44.00995
Rare Gases	0.003	0.002	---

Rare Gases	Ppm by volume	Ppm by weight	Molecular weight*
Neon	18.18	12.67	20.183
Helium	5.24	0.724	4.0026
Krypton	1.14	0.295	83.80
Hydrogen	0.5	0.035	2.01594
Xenon	0.087	0.394	131.30
Methane †	2.0	1.108	16.04303
Nitrous Oxide	0.5	0.760	44.0128

* On basis of carbon ¹² isotope scale (One Mole Air = 28.9644 gms.)

† The content of these gases may undergo significant variations from time to time or from place to place relative to the normal indicated for those gases.

The major constituents of air are nitrogen, oxygen, and argon. Carbon dioxide and the rare gases are present in a very small percentage. However, it will be shown that although helium and neon have very small concentrations, they play a significant role in the cryosorption of air in the pressure range of 10^{-4} torr.

For this study, it will be assumed that the atmospheric air at KSC is 77°F, the relative humidity is 80%, and the pressure is one atmosphere (760 torr). The water partial pressure at these conditions is ≈ 19 torr or 2.5 volume percent. Thus, the atmospheric air at KSC will be 2.5 volume percent water and the remainder will have the percentage given in table 8-1. It will also be assumed that the air leaking into the vacuum space has the same relative humidity as the air outside.

The equilibrium sorption isotherms shown in figures 7-2 and 7-3 will be used to determine the sorption capacity of the sorbents for the various gases, and it will be assumed that nitrogen, oxygen, and argon have approximately the same sorption characteristics. Cryosorption of the above gases at various temperatures is similar for coconut charcoal and molecular sieves. The sorption capacity may vary slightly. It will also be assumed, unless otherwise stated, that the sorbents are activated and have less than 1 weight percent pre-sorbed water.

The vapor pressures of water and the various constituents of air at temperatures down to 20 K become very important in a cryosorption application where it is necessary to maintain pressures $\leq 10^{-4}$ torr. The vapor pressures of water and the gases under consideration to pressures of 10^{-11} torr are given in figure 8-1. At 20 K (the approximate temperature of the liquid hydrogen line), the cryosorption of the

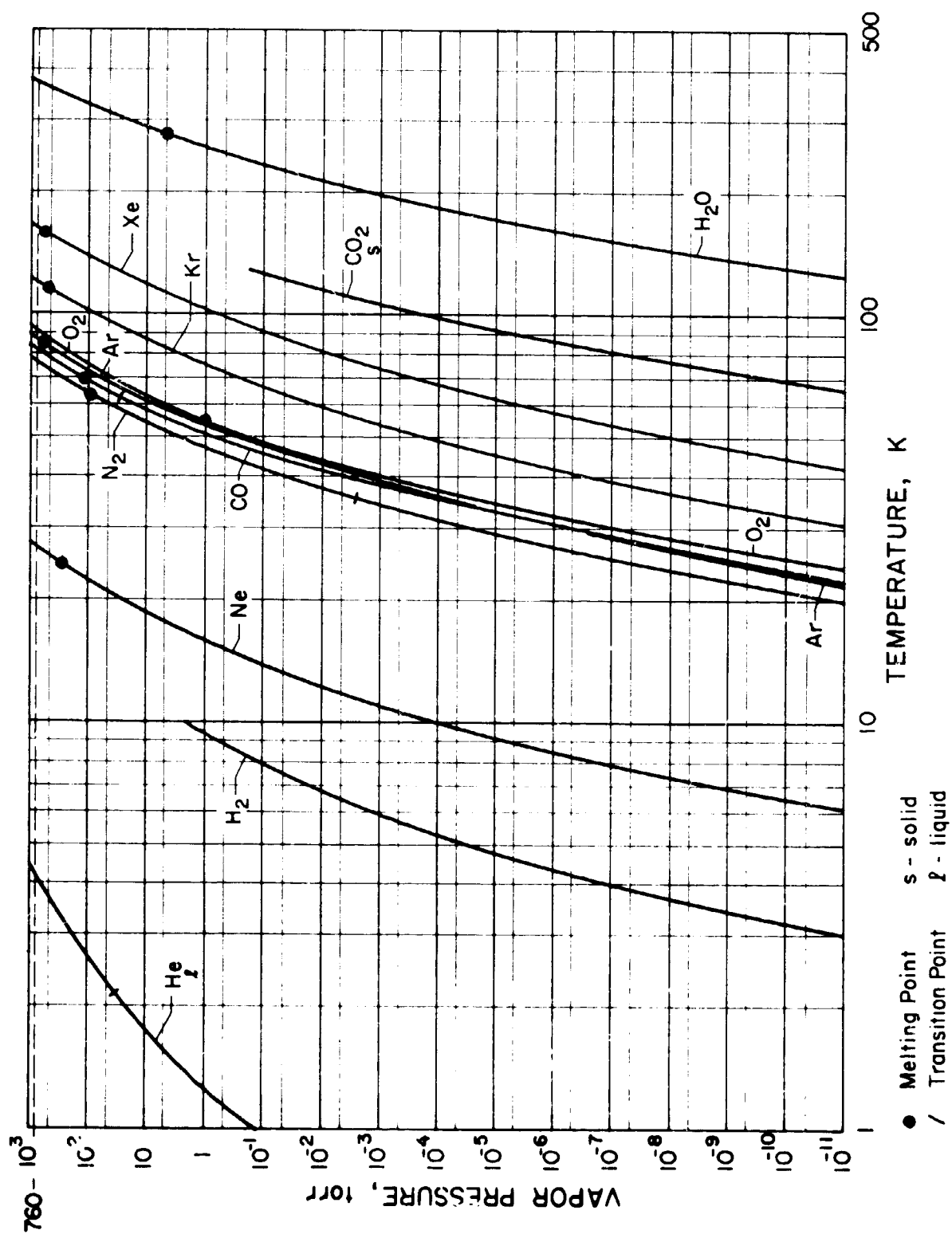


Figure 3-1 Vapor Pressures of the Atmospheric Gases and Water.

various constituents of air is not too much of a problem. All the gases, except neon, hydrogen, and helium, are condensed to the solid phase with vapor pressures less than 10^{-11} torr. The entire area of the inner line becomes a 20 K surface, in addition to the large surface area of the sorbent. At 20 K, both molecular sieves and charcoal have a strong attraction for hydrogen and neon, but helium is only weakly sorbed. Helium is not too much of a problem with cryosorption of air, since it is only present in 5 ppm concentration. So it is possible to very effectively sorb most of the constituents of air at 20 K and easily achieve a good insulating vacuum. At 90 K (the approximate temperature of the LOX lines), cryosorption of air is much more difficult. All of the constituents of air (except water) have vapor pressures above 10 torr, which means that these gases have to be sorbed to remove them from the vacuum space. At 90 K, nitrogen, argon, carbon dioxide, and oxygen are strongly sorbed by either molecular sieves or charcoal, but helium, hydrogen, and neon are very weakly sorbed. Recent experimental work by Stern [1969] on the cryosorption of air indicates that helium and neon, for all practical purposes, are not sorbed at all at 77 K. Thus, it can be seen that cryosorption of air at 90 K to achieve an insulating vacuum is much more difficult than at 20 K.

The ultimate attainable pressure is governed by the initial pressure within the vacuum space. If it is assumed that all of the nitrogen, oxygen, carbon dioxide, hydrogen, and argon are sorbed and none of the helium and neon are sorbed, a plot of initial pressure versus final residual pressure for a given vacuum space volume is shown in figure 8-2. The final temperature of the neon and helium is assumed to be 195 K, the average temperature between the 90 K and 300 K walls.

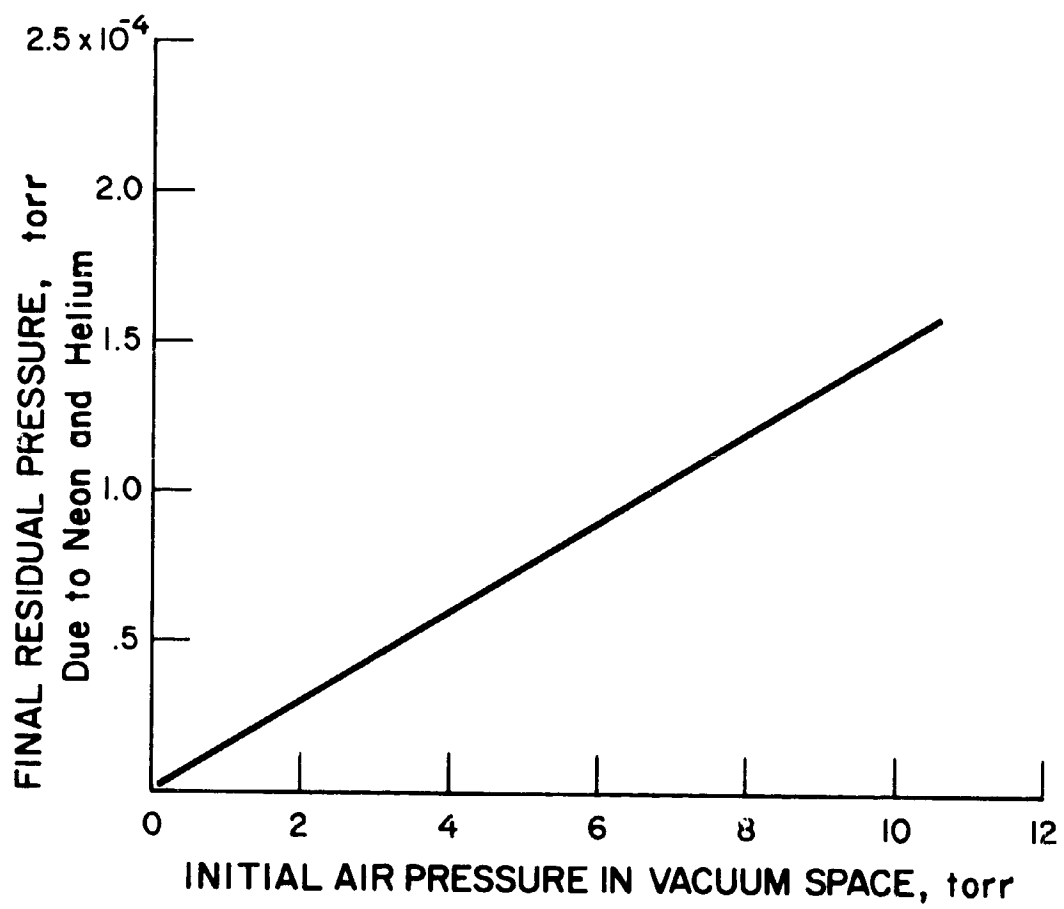


Figure 8-2 Residual Pressure due to Neon and Helium.

For an initial ambient pressure of 4000 microns, the residual gas pressure at 90 K would be 6×10^{-5} torr. This is within the range of a good insulating vacuum, but is not very much below 10^{-4} torr, above which the apparent thermal conductivity of multi-layer insulation begins to increase rapidly. There are also the possibilities of significant amounts of hydrogen being present due to metal outgassing, reduced sorption capacity due to presorbed water, and the possibility that all of the other gases are not sorbed. In view of this, it appears very possible that the residual pressure at 90 K could be $> 10^{-4}$ torr.

Stern [1969] recently conducted some very applicable experiments on the cryosorption of air, nitrogen, and argon with molecular sieve at 77 K. Figure 8-3 demonstrates the effect of helium and neon on the ultimate pressures that may be attained at 77 K.

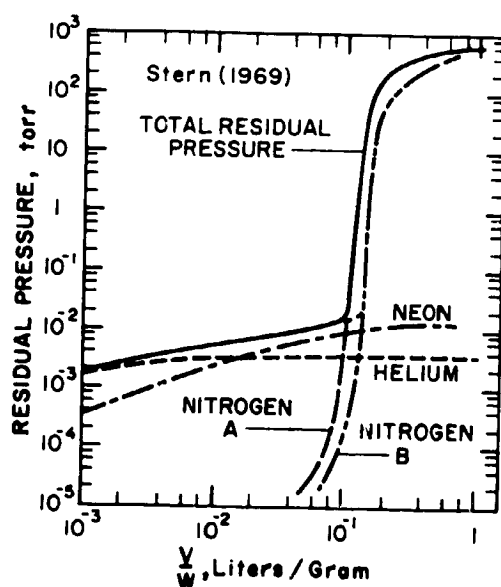


Figure 8-3 Residual Gas Pressures after Cryosorption at 77 K.

The residual nitrogen, neon, and helium pressures and the total residual pressure are given as a function of the V/W ratio (the vacuum space volume/sorbent weight ratio). The initial nitrogen pressure for curve A was 760 torr, and 594 torr for curve B. The initial neon pressure was 13.8×10^{-3} torr and the initial helium pressure was 3.08×10^{-3} torr.

The data clearly indicate the effect of neon and helium on air cryosorption at 77 K. The V/W ratios for the 10-inch hydrogen and 6-inch oxygen lines are 0.2 and 0.1, respectively. These numbers appear high when looking at the figure, but it must be remembered that the transfer lines were not designed to cryosorb air from one atmosphere initial pressure. The total residual pressure curve moves to the right for decreasing initial pressures. From the figure, it can be seen that helium is very weakly sorbed even at very low V/W ratios, and the initial partial pressure decreases very little. Neon is sorbed slightly more than helium, and at the lower V/W ratios, some of the neon is sorbed. This figure clearly illustrates the pressure limiting role of helium and neon when cryosorbing air at 77 K.

This figure also reveals two other very important considerations.

- 1) Air should never be used to pressurize a vacuum space that subsequently must maintain an insulating vacuum. It is much better practice to use nitrogen. Purging, or a few pressurizing cycles with nitrogen, also helps to remove the helium and neon if air has been admitted to the vacuum space.
- 2) If a vacuum space has been exposed to helium gas during a leak checking procedure using a helium mass spectrometer type leak detector, it is most important that the helium be removed from the vacuum space. This is very difficult when glass paper is used between the aluminum foil or aluminized Mylar. The space should

be repeatedly pressurized with nitrogen gas and evacuated to remove the residual helium gas. Helium is sorbed very weakly at 20 K, and for all practical purposes, it is not sorbed at all at 90 K.

8.2 Determination of Maximum Allowable

Ambient-Temperature Vacuum-Pumpout Level

One of the primary objectives of this study is to determine the maximum allowable vacuum-pumpout level that will permit the residual gas inside the vacuum spaces to cryopump to $\leq 10^{-4}$ torr pressure, when the transfer line is cooled to 20 or 90 K. In this section, the cryosorption characteristics of the sorbents at 20 and 90 K for cryopumping various gases to pressures $\leq 10^{-4}$ torr will be considered, to determine what ambient-temperature pumpout level is permissible. For this evaluation, the following assumptions will be made:

- 1) The residual gas in the transfer line vacuum spaces, which show no signs of excessive leakage and are mass spectrometer tight, is mostly hydrogen, due to outgassing from the metal.
- 2) Atmospheric leakage into the vacuum spaces is 80% relative humidity air at 77° F.
- 3) The sorbents were attached to the inner lines with very good thermal contact so the sorbent will quickly assume cryogenic temperature.
- 4) The gas sorbed by the sorbents at ambient temperatures will be considered negligible. The presorbed water cannot be considered negligible.
- 5) The sorbents are activated and the transfer line was fabricated using acceptable high-vacuum techniques.
- 6) Ideal gas relationships will be used for calculating pressure, temperature, and volume for the various cases.

7. The inner line of all spool sections is continuous tubing with no welds, and no hydrogen or oxygen gas leaks into the vacuum space from the inner line.
8. All sorption calculations are based on the pertinent parameters (volume, quantity of sorbent, etc.) of one typical 45-foot long spool-section vacuum space of that particular type of transfer line.

The equilibrium sorption capacity of the sorbents is given in terms of STP cm^3/gram . When calculating the sorption capacity of the sorbent for the various gases present in the vacuum spaces, the equilibrium sorption capacity of the respective pure gases at 10^{-4} torr pressure will be used to determine how much sorbent would be required to sorb all of that particular gas. In reality, each of the constituent gases of air has a respective partial pressure, and the entire amount of sorbent is sorbing the various gases as the sorbent cools. There is obviously some sorption interaction among the various gases on a given amount of sorbent, but since equilibrium sorption isotherms for the pure gases is the most popular method of presenting sorption capacity, these isotherms will necessarily have to be used to calculate the amount of sorbent required to cryosorb the various constituents of air at a given set of conditions. In order to evaluate the cryosorbing ability of the sorbents under various operating conditions, the following three typical transfer line vacuum space conditions will be considered.

Case 1: Very good transfer line section--slight pressure increase (50 - 1000 microns) due to normal hydrogen outgassing. Possibly as much as 4000 microns (worst case) over a long period of time. It will be assumed that the gas is mostly hydrogen from metal outgassing.

Case 2: Small leak in the outer line or annular end bellows allowing atmospheric air and moisture to leak into the vacuum space; pumping may be required every 90 - 120 days with vacuum space pressures increasing to as much as 10 torr before being pumped.

Case 3: Large leak in vacuum jacket; atmospheric air building to pressures near one atmosphere before being pumped; the pressure increasing in this manner possibly 2 - 3 times before leak is repaired. Transfer line space possibly open to atmospheric air for an arbitrary 8 hours during the repair period. It will be assumed that the leak has been repaired.

The above three cases will be evaluated for the different type sections of transfer line at their respective operating temperatures.

8.2.1 10-Inch Vacuum Jacketed Liquid Hydrogen Transfer Lines (Complex 39A)

Specifications: Sorbent → molecular sieve type 5A
Hydrogen getter → palladium monoxide
Multi-layer insulation → aluminum foil and glass paper
Operating temperature → 20 K
More complete specifications may be found in section 4.

Case 1: No external leak, assume the vacuum space pressure has risen to 4000 microns due to normal outgassing of hydrogen gas from metal and other vacuum space components. Because of the presence of palladium monoxide in these vacuum spaces (39A), it is unlikely that hydrogen gas would build to 4000 microns pressure during the 4-year period. However, it will be assumed that most of the gas is

hydrogen, and it will be determined how much activated molecular sieve would be required to sorb all of this hydrogen. From figure 7-3, the equilibrium hydrogen sorption capacity of molecular sieve 5A at 20 K and 10^{-4} torr is $180 \text{ cm}^3/\text{gram}$. At 4000 microns pressure inside the $230,597 \text{ cm}^3$ vacuum space, there are

$$\frac{4}{760} \times \frac{273}{300} \times 230,597 \text{ cm}^3 = 1225 \text{ cm}^3 [\text{STP}]$$

of gas to be sorbed. Assuming equilibrium sorption capacity, the amount of molecular sieves required to sorb all this gas would be

$\frac{1225 \text{ cm}^3}{180 \text{ cm}^3/\text{g}} = 6.8 \text{ grams}$. It was assumed that these vacuum spaces contain 1000 grams of activated molecular sieve, which is more than 100 times the amount required to sorb the 1225 cm^3 of hydrogen gas. Hence, there appears to be no problem in cryopumping the vacuum space to a good insulating vacuum when there is up to 4000 microns of hydrogen gas pressure inside the vacuum space at ambient temperature.

Case 2: For this case, we will first consider the sorption capacity of the molecular sieves at 77 K for the bulk of the gas--nitrogen, oxygen, and argon. There appears to be no sorption data available below 77 K for these gases. It will be further assumed that the sorption capacity of molecular sieves for oxygen and argon is the same as that of nitrogen. This is a reasonable assumption and is probably more accurate than using the small amount of sorption data for oxygen at low pressure. From figure 7-3, the equilibrium sorption capacity of molecular sieves for nitrogen at 77 K and 10^{-4} torr pressure is $85 \text{ cm}^3/\text{gram}$ of sorbent. At 4000 microns pressure, there are $\approx 1225 \text{ cm}^3$ [STP] of the above gases to be sorbed. Under ideal conditions, this would require

$\frac{1225 \text{ cm}^3}{85 \text{ cm}^3/\text{gm}} = 14.4 \text{ grams}$ of activated molecular sieve. Although conditions are not ideal, there are 1000 grams of molecular sieve in the vacuum space, which is considerably more than required to sorb these gases at 77 K. So, at this intermediate cooldown temperature of 77 K, there is ample capacity to sorb the nitrogen, oxygen, and argon. However, this transfer line operates at 20 K, and at this temperature, the entire area of the inner line becomes a cryosorption surface, and argon, nitrogen, and oxygen readily condense to solid at 20 K with a vapor pressure of $\leq 10^{-11}$ torr, as indicated in figure 8-1. Hence, there is very ample cryosorption capacity to sorb these three gases. The other gases of importance are neon, helium, and hydrogen. The relative abundance of hydrogen in atmospheric air is ≈ 0.5 ppm. There conceivably could be much more than this, due to metal outgassing, but as shown in case 1, the molecular sieves at 20 K have tremendous sorption capacity for hydrogen. Helium is present in amounts of 5 ppm in atmospheric air, and hopefully, there would be no additional helium present due to leak checking procedures. For a total atmospheric pressure of 4000 microns, the helium partial pressure is 2×10^{-5} torr. The sorption isotherm for helium on molecular sieves at 20 K and 2×10^{-5} torr gives an equilibrium sorption capacity of $6 \times 10^{-4} \text{ cm}^3/\text{gram}$. This figure is relatively low, but the amount of helium present in the vacuum space at 4000 microns air pressure is only 0.006 cm^3 .

A calculation reveals that it takes only $\frac{6 \times 10^{-3} \text{ cm}^3}{6 \times 10^{-4} \text{ cm}^3/\text{gm}} = 10 \text{ grams}$ of molecular sieve to sorb all the helium present in the vacuum space. Thus, there is ample sorption capacity to sorb the helium.

Neon is present in dry atmospheric air in concentrations of 18 ppm. At 4000 microns total pressure, the neon partial pressure is

$$\frac{18}{10^6} \times 4 \text{ torr} = 72 \times 10^{-6} \text{ torr} = 7.2 \times 10^{-5} \text{ torr},$$

which is already within the range of a good insulating vacuum. However, we also have the ability to sorb the neon too. Since there appears to be no equilibrium sorption isotherms for neon on molecular sieves at 20 K, it will be assumed that the sorption capacity is the same as neon on charcoal at 20 K. For a neon partial pressure of 7×10^{-5} , the molecular sieves at 20 K would have an equilibrium sorption capacity of $7 \text{ cm}^3/\text{gm}$. The amount of neon inside the vacuum space at 4000 microns pressure is $\frac{18}{10^6} \times 1225 \text{ cm}^3 = 22 \times 10^{-3} \text{ cm}^3$.

Since each gram of molecular sieve at 20 K would have the capacity to sorb 7 cm^3 of neon at a partial pressure of 7×10^{-5} torr, there is definitely no problem in sorbing the neon.

So, the calculations reveal that approximately 25 grams of activated molecular sieve would be saturated in cryosorbing air at 4000 microns to a pressure of 10^{-4} torr. This leaves 975 grams of molecular sieve in reserve (assuming ideal cryosorption conditions). So, there appears to be ample capacity to cryosorb air at 4000 microns to a pressure $\leq 10^{-4}$ torr.

The amount of water entering the vacuum space along with the atmospheric air must also be considered. It was arbitrarily established that the vacuum space might leak up to a pressure of 10 torr before being pumped. The water content of saturated air at 77°F and 760 torr is 2×10^{-2} grams of water [Union Carbide-a, 1970].
gram of dry air

The water content of 80% saturated air at 77°F would be 80% of that value, or 1.6×10^{-2} grams of water per gram of dry air. At 10 torr, the amount of air leaking into the vacuum space would be $\frac{10}{760} \times 230,597 \text{ cm}^3 = 3034 \text{ cm}^3$. The partial pressure of water at the above conditions is 19 torr, so there is $\frac{760 - 19}{760} \times 3034 = 2958 \text{ cm}^3$ of dry air inside the vacuum space. The density of dry air at 77°F and 760 torr is $1.1845 \times 10^{-3} \text{ gm/cm}^3$, and the dry air weight inside the vacuum space is $1.1845 \times 10^{-3} \text{ gm/cm}^3 \times 2958 \text{ cm}^3 = 3.504 \text{ grams}$. So, the vacuum space collects 3.5 grams of dry air each time the pressure rises to 10 torr. The amount of water accompanying the dry air is $\frac{1.6 \times 10^{-2} \text{ gms of H}_2\text{O}}{\text{gm dry air}} \times 3.5 \text{ gms dry air} = .06 \text{ gms water}$. So, 0.06 grams of water are collected in the vacuum space each time the pressure in the vacuum space rises to 10 torr due to atmospheric leakage. It will be assumed that all of this water is sorbed by the molecular sieves. It will next be determined how many times this can happen until the molecular sieves sorb 2 weight percent of water, at which point, the sorption capacity begins to decrease, as shown in figure 7-7. Two weight percent of 1000 grams of sorbent is 20 grams. At 0.06 grams per pressure cycle to 10 torr, the sieves would reach the 2 weight percent after 333 cycles. Pressure cycles to 10 torr are very likely to occur if a small leak is present. However, this calculation indicates that in this particular case, the molecular sieves are good for many pressure cycles to 10 torr before presorbed water becomes a serious consideration.

To sum up the cryosorption situation for case 2, there is an ample amount of molecular sieve to cryosorb air at 4000 microns pressure to an insulating vacuum of $\leq 10^{-4}$ torr, provided the sorbent is activated. The presence of an additional amount of hydrogen due

to metal outgassing is of no consequence. There is also ample sorption capacity for hydrogen. The moisture leaking into the vacuum space at 10 torr pressure is a very small amount and presents no serious problem in this case.

In a situation of this type, assuming the vacuum space requires pumping every 90 to 120 days, the cost of pumping at this time interval must be weighed against the cost of repair. It may be that periodic pumping would be much less costly than repairing the vacuum leak, especially if the molecular sieves were exposed to the atmosphere for any length of time. This would require a high temperature vacuum-bakeout to desorb the water.

Case 3: For this case, it will be assumed that the leak has been repaired, and the initial pressure before cooldown is 4000 microns. Cryosorbing air at 4000 microns pressure is easily done if the molecular sieves are properly activated and contain very little presorbed water. This situation was just evaluated in case 2. The most important consideration in case 3 is the effect of the presorbed moisture on the sorption capacity of the molecular sieves. The amount of water vapor leaking into the vacuum space each time the pressure rises to 760 torr will be calculated. At 80% relative humidity, the partial pressure of water at 77° K is ≈ 19 torr. The volume of the vacuum space is $230,597 \text{ cm}^3$. It will be assumed that there is $760 - 19 = 741$ torr partial pressure due to dry atmospheric air, or there is $\frac{741}{760} \times 230,597 \text{ cm}^3 = 224,832 \text{ cm}^3$ of dry air inside the vacuum space. At 77°F and 80% relative humidity, the ratio of water to dry air is 1.6×10^{-2} gms water/gm dry air. The density of dry air at 77°F and 760 torr is $1.1845 \times 10^{-3} \text{ gm/cm}^3$, and there are $1.1845 \times 10^{-3} \text{ gm/cm}^3 \times 224,832 \text{ cm}^3 = 266$ grams of dry air inside

the vacuum space. The amount of water is $\frac{1.6 \times 10^{-2} \text{ gms H}_2\text{O}}{\text{gm dry air}} \times 266 \text{ gms}$ of dry air = 4.26 gms H₂O. So, the molecular sieves are exposed to 4.3 grams of water every time the vacuum space pressurizes to 1 atm at 77°F, and it will be assumed that all of this water is sorbed by the sieves.

At 4.3 grams of water per cycle and the assumed 1000 grams of molecular sieves, the water sorbed per cycle is 0.43 weight percent. It is reasonable to assume that a vacuum space with a leak of this type might leak to near atmospheric pressure 2 - 3 times before being repaired and then be exposed to atmospheric conditions for several hours during repair. With this type of exposure, it would be easy to sorb 2 weight percent water. A leak of this magnitude should definitely be repaired as soon as possible. During five pressure cycles to 1 atmosphere, the sieves would sorb ≈ 2 weight percent water and the sieves would begin to lose their rated sorption capacity for other gases.

8.2.2 10-Inch Vacuum Jacketed Liquid Hydrogen Transfer Lines (Complex 39B)

Specifications: Sorbent \rightarrow coconut charcoal

Multi-layer insulation \rightarrow aluminized Mylar

Operating temperature = 20 K

No palladium monoxide getter

Case 1: Referring to figure 7-2, it can be seen that the sorption capacity of coconut charcoal at 20 K and 10^{-4} torr for hydrogen is 250 cm³/gram charcoal. Charcoal has very high sorption capacity for hydrogen at 20 K and the capacity is still very high at 10^{-6} torr pressure. At 4000 microns pressure inside the vacuum space, there is $\frac{273}{300} \times \frac{4}{760} \times 254,162 \text{ cm}^3 = 1218 \text{ cm}^3$ [STP] of hydrogen gas to be

sorbed. This would require $\frac{1218 \text{ cm}^3}{250 \text{ cm}^3/\text{gm}} \approx 5$ grams of activated coconut charcoal, assuming ideal conditions. With 1058 grams of charcoal inside the vacuum space, there is ample sorbent capacity for hydrogen at the 4000 micron initial pressure level. The sorption of hydrogen with charcoal at 20 K presents no problem.

Case 2: The cryosorption problem here is very similar to case 2 for the transfer line spaces containing the molecular sieves. It will be assumed that most of the gas in the vacuum space is air. The air could be rich in hydrogen due to metal outgassing, but hydrogen is sorbed very well at 20 K. Nitrogen, oxygen, and argon are sorbed very strongly by charcoal at 77 K. The sorption capacity of charcoal is similar to that of molecular sieves for these gases. For a similar situation with molecular sieves, the calculation indicated that ≈ 15 grams of molecular sieves would be required to sorb these gases at 77 K. A similar amount of coconut charcoal would sorb the gases just as well. As explained before, the inner line at 20 K becomes a cryosorbing surface for these gases. Oxygen, nitrogen, and argon condense to solid with a vapor pressure less than 10^{-11} torr on a 20 K surface. So, there is considerable cryosorbing capacity for these gases at 20 K. Cryosorbing the neon and helium present in atmospheric gas is also fairly easy at 20 K. Previous calculations for molecular sieves indicate that 11 grams of sorbent are required for the helium and neon, and the charcoal would be equally as good at cryosorbing these gases.

The water leaking into the vacuum space should also be considered. The moisture inside the vacuum space would be sorbed very weakly by the charcoal at these low pressures. The sorption capacity of charcoal for water at low pressures and ambient temperatures is very low as shown in figure 7-4. However, upon cooling, the water

would soon be frozen solid with a vapor pressure less than 10^{-11} torr as indicated in figure 8-1. From the above analysis, it is apparent that the cryosorption of the atmospheric gases and the small amount of water in this case present no problem.

Case 3: For this case, it will be assumed that the leak has been repaired, and the initial ambient pressure is 4000 microns. This case is similar to the previous situation considered, except there is the chance of sorbing much more water. Referring to figure 7-4, it can be seen that the sorption capacity of coconut charcoal at 20°C for water at a water partial pressure of 19 torr (1 atm total pressure) is very high (400 milligrams H_2O /gram of charcoal) or 40 weight percent. The charcoal could conceivably sorb this much water depending on the number of times the vacuum space pressure increased to one atmosphere and the length of time the sorbent was exposed to the atmosphere during repair. However, the sorption curve also indicates that the sorption capacity drops off drastically at the lower pressures. If the leak were repaired, and the vacuum space pumped to the standard 10 - 50 microns at ambient temperature, most of the water would be desorbed from the charcoal, leaving the sorbent in a near activated condition with very little presorbed water. There is a slight hysteresis effect when desorbing water as explained in section 7.3.1. Conceivably, it could take a long time to desorb the water from the charcoal at concentrations of 40 weight percent, but the important point is that the charcoal can be activated at ambient temperatures. Elevated temperatures are not required. After desorption, the charcoal would have near rated capacity for the other gases. It was shown in the previous case 2 that there is plenty of sorption capacity for air with the charcoal and transfer line at 20 K. Hence,

there should be no difficulty in cryosorbing the gases to a good insulating vacuum in this case, and the moisture should present no problem.

8.2.3 6-Inch Vacuum Jacketed LOX Transfer Lines
(Complex 39A and B)

Specifications: Sorbent → molecular sieve 5A

Multi-layer insulation → aluminum foil and glass paper

Hydrogen getter → palladium monoxide present in the vacuum spaces of both 39A and B.

Operating temperature → ~ 90 K

Case 1: Palladium monoxide is present in all the vacuum spaces of the 6-inch LOX transfer lines (39A and B). This should convert the hydrogen gas to water, which is then strongly sorbed by the molecular sieves, as explained in section 5 of this report. However, the worst case will be assumed, i. e. , most of the gas is hydrogen, and the initial pressure is 4000 microns. Referring to figure 7-3 to determine the sorption capacity of molecular sieves at 90 K, it becomes apparent that there is no sorption data for hydrogen at 90 K in this pressure range. However, if an extrapolated isotherm is extended between the data of Bailey and the Union Carbide isotherm to lower pressures, it appears that the sorption capacity of molecular sieve 5A at 77 K would be near $10^{-2} \text{ cm}^3/\text{gram}$ at a pressure of 10^{-4} torr. Checking this number with the sorption capacity of charcoal, to determine if this capacity is near that of charcoal, we find that the sorption capacity of charcoal at 80 K and 10^{-4} torr is exactly $10^{-2} \text{ cm}^3/\text{gram}$. This value will be used, but it is probably high because the operating temperature of the LOX lines is 90 K or higher, and the sorption capacity of all sorbents for hydrogen is very temperature dependent. At 4000 microns pressure, there is $\frac{4}{760} \times \frac{273}{300} \times 157,512 \text{ cm}^3 = 753 \text{ cm}^3$ [STP] of

hydrogen gas to be sorbed, and it would require

$\frac{753 \text{ cm}^3}{10^{-2} \text{ cm}^3/\text{gm}} = 75,300$ grams of molecular sieves to sorb the hydrogen gas at 10^{-4} torr pressure. There are only 1600 grams of molecular sieve in each spool-section vacuum space. Sixteen hundred grams of molecular sieve are obviously not enough sorbent to cryopump this space to 10^{-4} torr. This calculation vividly points out the effect of the four orders of magnitude difference in the hydrogen sorption capacity of these sorbents at 20 and 90 K.

This obviously is the worst condition to be encountered. If it is assumed that a vacuum space pressure of 10^{-3} torr is acceptable, it appears that the sorption capacity of the sieves at this pressure would increase by a factor of 5 - 10. If a factor of 10 is assumed, this still means 7,500 grams of sorbent would be required. If it is further assumed that the 4000 micron level is obviously too high, the sorption characteristics at 1000 microns initial pressure should be considered. At 1000 microns pressure, there is $\frac{1}{760} \times \frac{273}{300} \times 157,512 \text{ cm}^3 = 189 \text{ cm}^3 [\text{STP}]$ of hydrogen gas to be sorbed, and it would require

$\frac{189 \text{ cm}^3}{10^{-2} \text{ cm}^3/\text{gm}} = 18,900$ grams of molecular sieve to sorb this much hydrogen. This is still considerably more sorbent than is present inside the vacuum spaces.

If a more realistic situation is assumed, i. e., because of the palladium monoxide, only 10% of the gas at the 1000 micron pressure level is hydrogen, the amount of sorbent required would be

$\frac{18.9 \text{ cm}^3}{10^{-2} \text{ cm}^3/\text{gm}} = 1890$ grams of charcoal. This is still more sorbent than is available, and the other 90% of the gas also has to be sorbed. If the 10^{-4} vacuum level is then reduced to 10^{-3} torr, the sorption capacity may increase by as much as a factor of 10; in which case, 189 grams of sorbent would be required to sorb the hydrogen gas under ideal conditions.

From these calculations, it is quite evident that the cryosorption of hydrogen at 90 K is extremely difficult, and very little hydrogen can be tolerated in the vacuum space. For this reason, it is very important to reduce the hydrogen outgassing of the metal to a minimum by giving the vacuum spaces a good hydrogen bakeout, as explained in section 4. Moreover, it is also very important to place palladium monoxide inside the vacuum spaces of transfer lines that operate at 90 K. The virtues and characteristics of palladium monoxide were explained in section 4.

Case 2. It was previously determined that the moisture leaking into the 254 liter 10-inch hydrogen vacuum spaces was ≈ 0.06 grams/pressure cycle to 10 torr; the air being 80% saturated with moisture. The calculation revealed that the vacuum space could be pressurized to 10 torr for approximately 333 times before the 1000 grams of molecular sieve would sorb 2 weight percent of water.

Since the volume of the LOX vacuum spaces is only 158 liters, and this smaller volume contains even more sorbent (1600 grams), considerably more than 333 cycles would be required to bring in 2 weight percent water. Therefore, the moisture leaking into the vacuum spaces in this particular case presents no problem.

It will be assumed that the gas inside the vacuum space is air at 4000 microns pressure. It will also be assumed, as before, that the sorption capacity of molecular sieves at 90 K is approximately the same for argon, oxygen, and nitrogen. Referring to figure 7-3, the sorption capacity of molecular sieves at 77 K for nitrogen at 10^{-4} torr pressure is $85 \text{ cm}^3/\text{gram}$. The sorption capacity at 90 K is undoubtedly lower, but the 77 K figure will be used. Since most of the gas is nitrogen, argon, or oxygen, the $85 \text{ cm}^3/\text{gram}$ will be used to calculate the

amount of molecular sieves required to cryopump the vacuum space to 10^{-4} torr pressure. At 4000 microns, there are 753 cm^3 [STP] gas to be sorbed which would require $\frac{753 \text{ cm}^3}{85 \text{ cm}^3/\text{gm}} \approx 9$ grams of molecular sieve to cryosorb this gas to 10^{-4} torr pressure, assuming ideal conditions. So there appears to be ample capacity for cryosorbing these three gases at 90 K.

The cryosorption of neon and helium at 90 K will be considered. Referring to figure 7-3 again, to check the sorption capacity of molecular sieves at 90 K and 10^{-4} torr pressure for neon and helium, it is readily apparent that cryosorption of these gases at 90 K is very difficult. The sorption capacity of neon at 77 K is $10^{-5} \text{ cm}^3/\text{gram}$ at 10^{-4} torr pressure, and it is undoubtedly less than that at 90 K. The sorption capacity of helium at 77 K is $\approx 10^{-6} \text{ cm}^3/\text{gram}$ at 10^{-4} torr pressure, and it too is undoubtedly less at 90 K. Stern [1969] found from his recent work on the cryosorption of air on molecular sieves at 77 K that helium and neon were essentially not sorbed at all at 77 K. Hence, for this study, the assumption will be made that helium and neon are not sorbed at all on molecular sieves at 90 K. Referring to figure 8-2, it can be seen that the residual pressure at 90 K due to neon and helium in air at an initial pressure of 4000 microns is 6×10^{-5} torr. This is exceedingly close to 10^{-4} torr, when considering the possibility of a little hydrogen being present due to metal outgassing or a little additional helium being present due to leak checking operations. At an initial pressure of 1000 microns, the residual gas pressure due to neon and helium is 1.5×10^{-5} torr. This gives a more comfortable margin below the 10^{-4} torr pressure level. To sum up case 2, the moisture is no problem, but neon and helium are for all practical purposes only cooled and not sorbed, and the hydrogen is

only weakly sorbed. The inner line surface at 90 K is of no value for cryopumping helium, neon, and hydrogen, because none of these gases will condense on the inner line surface. Considering the above cryosorption capacities and characteristics, the 4000 micron pumpout level appears to be very marginal for the 90 K LOX transfer lines, but a 1000 micron pumpout level is reasonable.

Case 3: This case is similar to the previous situation with regard to the atmospheric gases (marginal), except there is the chance of sorbing much more water. The amount of water leaking into the vacuum space each time the pressure rises to 760 torr will be calculated. The vacuum space volume is $157,512 \text{ cm}^3$. At 80% relative humidity, the partial pressure of water at 77 K is ≈ 19 torr. It will be assumed that there is $760 - 19 = 741$ torr partial pressure due to dry atmospheric air, or there is $\frac{741}{760} \times 157,512 \text{ cm}^3 = 153,574 \text{ cm}^3$ of dry air. At 25°C and 80% relative humidity, the ratio of water to dry air is 1.6×10^{-2} grams water/gram of dry air. The density of dry air at 25°C and 760 torr is $1.1845 \times 10^{-3} \text{ gm/cm}^3$, and there are $1.1845 \times 10^{-3} \text{ gm/cm}^3 \times 153,574 \text{ cm}^3 = 182$ grams of dry air inside the vacuum space. The amount of water inside the vacuum space is $1.6 \times 10^{-2} \text{ gms H}_2\text{O}$ gm dry air $\times 182 \text{ gms dry air} = 2.9 \text{ grams H}_2\text{O}$. It will be assumed that the molecular sieves sorb this much water each time the vacuum space is pressurized to 1 atmosphere. There are 1600 grams of molecular sieve inside the vacuum spaces, so 2 weight percent of 1600 grams is 32 grams. At this rate, the vacuum space could pressurize to 1 atmosphere 11 times before the sieves sorbed 2 weight percent water. Hence, it appears that if the vacuum space leaks to 1 atmosphere 2 - 3 times as assumed, there will be no appreciable decrease in sorption capacity due to presorbed water, and the

molecular sieves will not have to be activated at elevated temperatures.

The amount of exposure to the open atmosphere at 80% relative humidity during repair will determine whether the sieves have to be activated at the recommended temperature. If the sorbent is exposed to the open atmosphere very long, as discussed in section 7, the molecular sieves should be activated to insure that they have rated sorption capacity.

8.3 Conductance and Cryopumping Rate

The flow of the gas within the vacuum spaces becomes important when considering cryopumping from higher initial pressures. Most of the gas must be sorbed by the sorbent, which is hopefully spaced at given intervals along the vacuum space section instead of at one central position. Thus, most of the gas has to flow along the annular space, or from within the interstitial spaces of the multi-layer insulation to the sorbent. If the inner line temperature is at 20 K, the path is not so tortuous, because a fair share of the condensibles (N_2 , O_2 , Ar, H_2O) would be condensed to solid all along the inner line. At 90 K, most of the residual gas would have to be sorbed by the sorbent.

The equilibrium sorption isotherms in figures 7-2 and 7-3 were obtained under ideal cryosorption conditions. One typical cryosorption apparatus was made of parallel discs thermally connected to the cryogen. The discs were well spaced with large holes in the center and approximately a 1/2-inch layer of sorbent on each annular disc. The apparatus had very good conductance to the sorbent, and most of the sorbent was near the cryogen temperature. The conditions inside the vacuum space of the transfer line containing multi-layer insulation are considerably different from the cryosorption apparatus.

The time required to cryopump a typical vacuum space from 4 torr to 10^{-4} torr is dependent upon flow conductance [Dushman, 1949], and is dependent upon system geometry, fabrication methods, etc. No attempt will be made in this study to determine this time interval because there are too many unknown variables to be considered. The cooling rate of the inner line, the condition and location of the sorbents, the mean temperature of the bulk of the sorbent, and the reduced conductance due to the presence of the multi-layer insulation are all variables of significant importance. If the time required to cryopump from 4 torr to 10^{-4} torr pressure is considered to be of major importance, it would be best to conduct an experiment using a typical section of vacuum insulated transfer line, or better yet to monitor the vacuum space pressure of several vacuum sections during a cooldown test of the actual transfer line.

9.0 Vacuum Gauge Study

Past performance of the thermocouple type probes and meters used for vacuum measurement at KSC has been marginal. Different battery-powered meters used to read the vacuum levels will give considerably different values when measuring the pressure inside a given vacuum space. There appears to be a calibration problem between the various meters and the probes. Also, the probes have experienced excessive corrosion of the pins and around the base of the pins, due to the high humidity and the saline environment at KSC. The corrosion causes poor electrical contact at the pins and also causes small holes at the base of the pins, resulting in leakage into the vacuum probe space, or in some cases, leakage into the main vacuum space. There has also been a problem of leakage around the probe threads and a fairly high probe failure rate due to vibration and shock incurred by the transfer lines. In addition to the above problems, the thermocouple type meters

presently used have an upper pressure limit of 1 torr and the vacuum readings near 1 torr are not too accurate. If the vacuum level is increased to 4 torr, the present thermocouple type meters with a pressure range of 1 torr will not be adequate.

Because of the above problems with the present vacuum measuring system, this study was initiated to survey the commercial vacuum measuring instruments available and determine if more suitable and reliable measuring instruments are obtainable for the KSC application.

9.1 Scope of the Vacuum Gauge Study

The scope of this study will be to consider and evaluate all vacuum instruments that could be used to measure the vacuum level in the transfer line vacuum spaces at KSC. Most of the basic type instruments in the proper operating range will be considered. The philosophy of the vacuum gauge study will be to point out the advantages and disadvantages of the basic type instruments, point out the characteristics pertinent to the KSC application, and suggest procedures in which they might be used to check the vacuum spaces. A specific type of instrument will not be recommended as a result of this study. This decision will be left to the personnel at KSC.

9.2 Assumptions and Criteria for Evaluation

The following assumptions were made for the vacuum gauge study:

- 1) It was assumed that the ambient temperature pumpout level for the liquid hydrogen system will be increased to 4000 microns, and the vacuum spaces, when pumped, are evacuated to 10 - 50 microns pressure. Hence, the pressure range for the vacuum measuring instruments under consideration is 10 to 4000 microns.

- 2) It will be assumed that the vacuum measurements will be made only when the transfer line is at ambient temperature, and that there is a good vacuum valve between all vacuum probes and the main vacuum space. It is understood that some vacuum sections do not have such a valve at the present time.
- 3) It will be assumed that there is 110 volt ac power available along the cross-country transfer lines to operate vacuum instruments and a small vacuum pump, or that other power sources are available.
- 4) It will be assumed that a small thermocouple-type threaded probe is not a prerequisite for consideration of a vacuum instrument. Instruments of various types and probe configurations will be evaluated for possible use.

The following criteria were used in evaluating vacuum measuring instruments:

- 1) The corrosive, high humidity saline atmosphere at KSC. Vacuum probe pins and electrical connectors permanently installed would be subject to this corrosive action.
- 2) The vibration and shock that a fixed probe incurs during transfer line cooldown and launch.
- 3) The reliability and repeatability of the measuring instrument.
- 4) The accuracy of the instrument. Extreme accuracy is not required for this application.
- 5) The pressure range under consideration. Reasonably accurate readings are required in the range from 10 to 4000 microns pressure.

- 6) The cost of the vacuum measuring system.
- 7) The simplicity of operation.
- 8) The desirability of using a portable, battery-powered meter to read vacuum levels.
- 9) The principle of operation, complexity of the instrument, and ease of maintenance.

9.3 Basic Types of Vacuum Gauges Considered

Using the above assumptions and criteria for evaluating the various vacuum instruments available, nine basic types of vacuum instruments were chosen for consideration. These are enumerated below.

- 1) Thermocouple or thermopile type gauges.
- 2) Pirani or thermistor type gauges.
- 3) Capacitance manometers.
- 4) McLeod gauges.
- 5) Molecular vacuum gauges.
- 6) Ionization gauge with a radioactive source.
- 7) Quartz Bourdon tube vacuum gauge.
- 8) Ionization - Pirani gauge combinations.
- 9) Dial gauges.

A list of typical various manufacturers of the above type gauges and their pertinent characteristics is given in table 9-1.

Table 9-1

Typical Vacuum Gauge Manufacturers

Type of Gauge	Manufacturer	Series Model	Pressure Range	Portable	No. of Stations	Battery Powered	Special Features	Dependent on Gas Composition
Capacitance Manometer ↓	MKS Instruments	77	10^{-5} to 1000 torr	Yes	One or more	No	Seven ranges available.	No
		90	10^{-5} to 1000 torr	Yes		No	High temperature (450° C) bakeout capability.	No
		100	10^{-5} to 1000 torr	Yes		No	Automatic digital readout, manual and auto rangings, multi-channel, high accuracy and linearity.	No
		126	10^{-5} to 1000 torr	Yes		No		No
		144	10^{-5} to 1000 torr	Yes		No		No
Ionization Gauge Using Radioactive Source	Granville-Phillips	212	5×10^{-3} to 1000 torr	Yes	1	No	Five ranges for 3 sensing heads.	No
		1014	10^{-5} to 1000 torr	Yes	1	No	Seven ranges, digital readout, high precision, wide range.	No
	Data Metrics Inc.	1018	10^{-5} to 1000 torr	Yes	1	No		No
		1023	10^{-5} to 5000 torr	Yes	1	No		No
		820	10^{-3} to 760 torr	Yes	1	No	100 mc Radium ²²⁶ Radioactive source, emits α and β particles.	Yes
Dial Type Pressure Gauges	Wallace & Tiernan	FA160150	0.1 to 20 torr	No	1	No	Not damaged by atmospheric pressure.	No
Thermistor or Pirani Type ↓	Kinney Vacuum	KTPG	to 50 torr	Yes	1 to 3	No		Yes
	General Electric	22GC310	10^{-3} to 760 torr	Yes	1	No		Yes
		22GC321	10^{-3} to 760 torr	No	2	No	Panel Mount.	Yes
	Bendix (Auto Vac)	3294-B	10^{-3} to 100 torr	Yes	1 to 4	No	Two scales, automatic ranging.	Yes
	Edwards High Vacuum		10^{-3} to 500 torr	Yes	1 to 4	---		Yes

Table 9-1 (Continued)

Typical Vacuum Gauge Manufacturers

Type of Gauge	Manufacturer	Series Model	Pressure Range	Portable	No. of Stations	Battery Powered	Special Features	Dependent on Gas Composition
Thermistor or Pirani Type ↓ Thermopile ↓ Thermocouple	Japan-Vacuum Engr. Co.	GP-2DH	3×10^{-3} to 20 torr	Yes	2	No	Dual scale.	Yes
	Leybold-Heraeus	PM-201	10^{-3} to 100 torr	Yes	1	No	Two-station model.	Yes
	Leybold-Heraeus	PM-202	10^{-3} to 100 torr	Yes	2	No		Yes
	Central Scientific	94-190-1	10^{-3} to 2 torr	Yes	1	No	Dual scale.	Yes
	Central Scientific	9472-3	10^{-3} to 2 torr	Yes	4	No	Dual scale.	Yes
	Pennwalt-Stokes	TB-3	to 1 torr	Yes	1 to 5	No	Portable with leather carrying case.	Yes
		TB-4	to 20 torr	Yes	1 to 5	No		Yes
		TB-5	to 10^{-1} torr	Yes	1 to 5	No		Yes
	Hastings-Raydist Inc.	TP7A	to 20 torr	Yes	1	Yes	Portable with leather carrying case.	Yes
		TV4A	to 1 torr	Yes	1	Yes	Portable with leather carrying case.	Yes
Thermocouple ↓	Fredricks Co.	VT6B	to 1 torr	Yes	1	No	Cabinet model.	Yes
		VT6S2	to 1 torr	Yes	2	No	Two-station model.	Yes
		VT6S	to 1 torr	Yes	5	No	Five-station model.	Yes
		VH3	to 5 torr	Yes	1	No	Compact-economical model.	Yes
		2AM	10^{-3} to 10^{-1} torr	Yes	1 to 6	Yes	Dual scales.	Yes
	Veeco	2AM	10^{-3} to 1 torr	Yes	1 to 6	Yes		Yes
		2AM	10^{-3} to 5 torr	Yes	1 to 6	Yes		Yes
	Veeco	TG-27	to 20 torr	Yes	1 to 3	No	Dual scales.	Yes
		TG-7	10^{-3} to 1 torr	Yes	1 to 3	No		Yes
	Picker Nuclear	VC-12	10^{-4} to 5 torr	Yes	1	No	Dual scales.	Yes
	Varian Assoc.	9720000	to 1 torr	Yes	1	No		Yes
		GMJ-100	10^{-3} to 1 torr	Yes	1 to 5	No		Yes
	Torr Vacuum Products	GMJ-100B	10^{-3} to 1 torr	Yes	1 to 5	Yes		Yes

Table 9-1 (Continued)

Typical Vacuum Gauge Manufacturers

Type of Gauge	Manufacturer	Series Model	Pressure Range	Portable	No. of Stations	Battery Powered	Special Features	Dependent on Gas Composition
Thermocouple	Norton Vacuum	801 804	to 2 torr to 2 torr	Yes Yes	1 5	No No		Yes Yes
	Kinney Vacuum	KTB-1	10^{-3} to 3 torr	Yes	1	Yes	Rechargeable N-C batteries.	Yes
Thermistor-Ionization Combination	Central Scientific	94178	10^{-3} to 1 torr	Yes	1	No		Yes
	Kinney Vacuum	KTG	10^{-3} to 3 torr	Yes	1 to 6	No		Yes
	Bendix	GTC	10^{-3} to 1 torr	Yes	1 or 2	Yes	Battery or ac power.	Yes
	Bendix	GMA 201	10^{-3} to 760 torr	Yes	1 to 4	No	Two scales.	Yes
	Kinney Vacuum	KITG	2×10^{-11} to 1 torr	No	1	No		Yes
	Granville-Phillips	260	2×10^{-10} to 1000 torr	No	1	No	Manual or auto ranging, automatic filament protection.	Yes
	Veeco	RG-81 TG-7	2×10^{-11} to 1 torr	No	1	No	One ion gauge and two T. C. connections.	Yes
	Veeco	DG2-2T	5×10^{-6} to 1 torr	No	1	No	One cold cathode and two TC connections.	Yes
	Kinney Vacuum	TD	10^{-3} to 150 torr	Yes	1	No	380 torr upper pressure limit.	No
	Mechanically Actuated McLeod Gauge							
Stokes Tilting McLeod Gauge	Pennwalt-Stokes	276-50 276-AA 276-BB 276-AG	10^{-2} to 50 torr 10^{-3} to 5 torr 10^{-4} to 0.5 torr 10^{-5} to 5 torr	Yes Yes Yes Yes	1 1 1 1	No No No No	Small field type McLeod gauge enclosed in plastic case.	No No No No
	Texas Inst.	144 145	to 259 torr to 259 torr	No No	One or more	No No	Very sophisticated and expensive.	No No
Quartz Bourdon Tube								

The principle of operation, the advantages, and the disadvantages of the nine vacuum measuring systems are given in the following sections.

9.3.1 Thermocouple or Thermopile Type Gauges

The thermocouple and thermopile probes are similar in operation. The thermocouple probe has a thermocouple spot welded to a resistive heating element, which is heated with constant electrical current. The residual gas cools the resistive element and attached thermocouple and operates on the principle that the heat transfer rate is proportional to the absolute pressure of the gas. The EMF from the thermocouple is read in units of pressure on a voltmeter. Most thermocouple gauges have an operating range of 10^{-3} to 1 torr. A plot of thermocouple current versus pressure is shown in figure 9-1. This plot shows that the optimum operating range is from 1 micron to 600 microns. The curve is almost flat in the range of 600 to 1000 microns and the sensitivity is very poor in this range. Some thermocouple instruments are offered with two scales, one being a separate range for the 1000-5000 micron region. The accuracy of the instrument in this higher pressure range is probably not as good as in the lower range, so a separate scale is certainly desirable. A dual scale meter with an upper pressure limit near 5000 microns should be given serious consideration if thermocouple probes are to be used in the pressure range to 4000 microns.

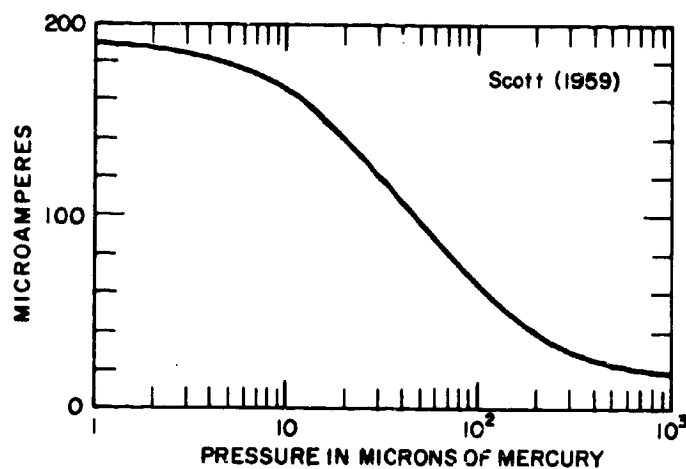


Figure 9-1 Calibration Curve for a Commercial Thermocouple Gauge.

The thermopile type probe is more sophisticated and some are temperature compensated. A typical temperature compensated circuit employs two thermocouples in series with an ac circuit. The array of thermopile elements are securely butt welded into continuous wires, which constitute the sensing element. A sketch of a thermopile sensor and the electrical circuit is shown in figure 9-2. The hot junctions of the thermopile are heated by the ac current, while the cold junctions are kept near ambient temperature by the heavy copper mounting studs. The thermopile is arranged so that the positive sides of thermocouple A and B face each other.

A third thermocouple (C) in the dc meter circuit compensates for changes in ambient temperature. The thermopile type probes have more

thermocouple junctions and more connections to heavy mounting studs and are undoubtedly more susceptible to damage from shock and vibration. However, the thermopile type probes do offer a little higher pressure range than the simple thermocouple probes. Several reliable companies offer special probes and matching meters with pressure ranges from 0 to 20 torr.

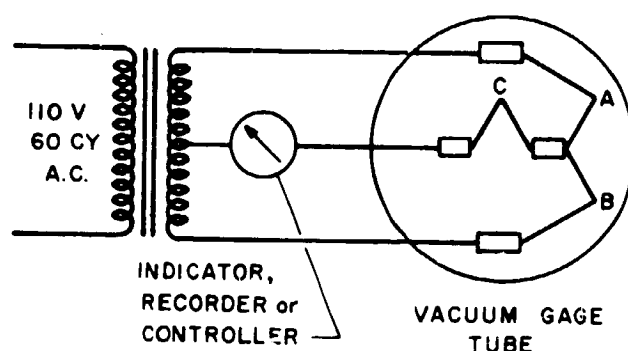


Figure 9-2 Thermopile Sensor and Electrical Circuit.

Thermocouple and thermopile sensors are comparatively low in price and are probably more rugged and less susceptible to vibration damage than any of the other type meters. The probes are not damaged by exposure to atmospheric pressure and overpressure is no problem. The gauge tubes are offered by several reliable manufacturers with gold-plated pins and noncorrosive stainless steel bodies. These type probes measure condensible and noncondensable gas pressure; however, both of these probes are sensitive to the type of gas being measured. This is one disadvantage of this type vacuum gauge.

A significant error can result in the pressure reading if the vacuum space contains hydrogen gas due to metal outgassing. Most of the meters are calibrated for air, and this calibration works well if air or nitrogen is leaking into the vacuum space. However, if the vacuum space is rich in hydrogen due to metal outgassing, an error factor of 2 - 3 in pressure measurement can result. Figure 9-3 shows the effect of measuring hydrogen gas instead of air, for which the probe is calibrated. In the pressure region of 1 torr, the difference in meter readings is not too great between air and hydrogen. However, at 4 torr, the error becomes appreciably greater. This type of error is not serious for the KSC application; it amounts to a vacuum space being pumped at 2.4 torr instead of 4 torr pressure. Also, most of the vacuum spaces except the LH_2 lines at Launch Complex 39-B contain palladium monoxide, which would tend to convert the hydrogen to water. If the vacuum space contains a high concentration of oxygen, possibly due to inner line leakage, the thermopile meter would give a pressure reading lower than the actual pressure. The error is not significant in the pressure range of concern; for example, a meter reading of 4 torr would be given for oxygen gas at 5.5 torr pressure.

Most of the secondary type vacuum measuring instruments are sensitive to the type of gas being measured. Secondary type instruments are those using a property of the gas such as thermal conductivity or viscosity to measure pressure, rather than actually measuring pressure directly. One would have to conclude, that for the KSC application, using a vacuum measuring system that is sensitive to the type of gas being measured is not a serious disadvantage. The thermocouple and thermopile type probes have the added advantage of being able to use battery-powered meters to read vacuum levels.

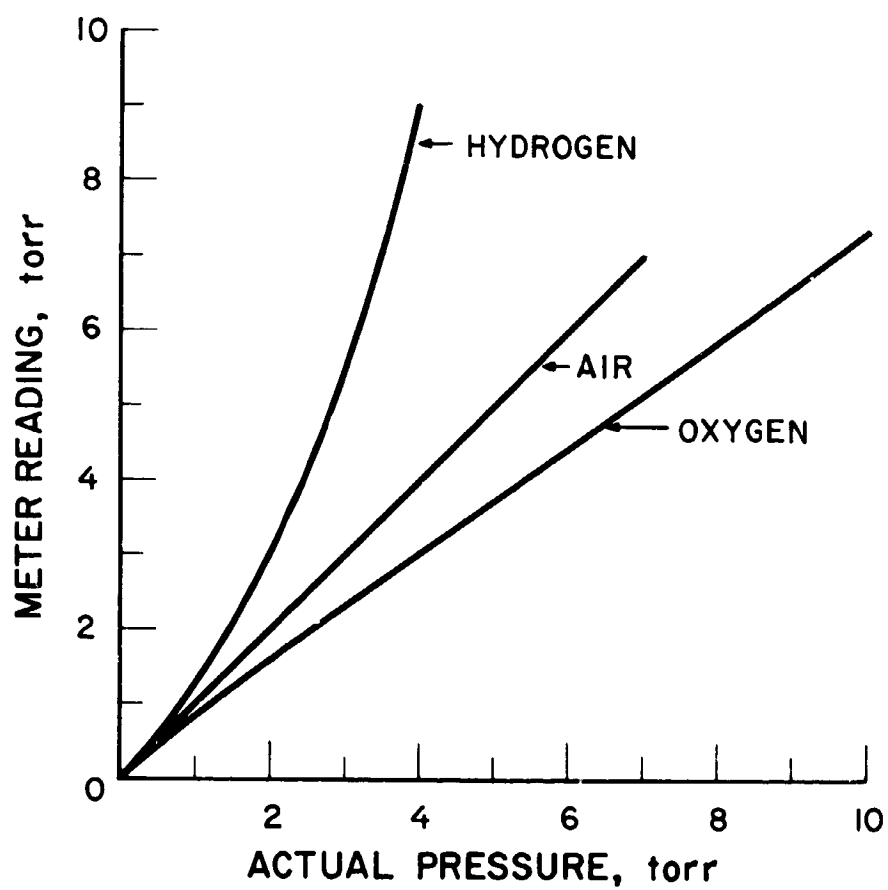


Figure 9-3 Calibration of a Typical Thermopile Type Vacuum Probe (Probe Calibrated for Air).

9.3.2 Pirani or Thermistor Type Gauges

The Pirani or thermistor type vacuum measuring system is similar in principle to the thermocouple and thermopile type gauges. These gauges have the capability of being considerably more accurate than the thermocouple or thermopile type gauges. A thermistor (very temperature sensitive resistor) is used as the pressure sensing element and also as one leg of a Wheatstone bridge circuit. A second identical thermistor is permanently sealed in an evacuated bulb. The exposed thermistor changes resistance as the residual gas tends to cool it. This vacuum instrument uses the same principle as the previous gauges, in that the heat transfer rate is a function of the absolute pressure of the gas. The Pirani gauge has an operating pressure range considerably higher (10^{-3} to ~ 100 torr) than the thermocouple type gauges. The Pirani gauge senses condensible and noncondensable gases, but again is sensitive to the type of gas being measured. The gauge probes are probably not as tolerant to vibration and shock as the thermocouple and thermopile type gauges because of the increased mass of the thermistors. These gauges can be made very accurate even in the higher pressure ranges.

9.3.3 Capacitance Manometers

A capacitance manometer is a primary pressure measuring device and is capable of very high accuracy. The sensing head is not a probe type that could be screwed into a vacuum probe space. One type of head is approximately 3 inches square x 3 inches high with two 1/8-inch female pipe threads for the pressure to be measured and the reference pressure. The sensing element separates the two chambers inside the sensing head and consists of a very thin stainless steel diaphragm. On each side of the diaphragm and in close proximity are two insulating

discs which also support two capacitive electrodes. A pressure differential across the diaphragm increases the capacitance of one electrode and decreases the capacitance of the other. The capacitors are connected to an electrical ac bridge circuit. The change in capacitance is calibrated in terms of pressure difference. The capacitance manometer has a very wide range from 10^{-5} to 1000 torr. The instrument measures condensible and noncondensable gases; it is not sensitive to gas composition; it is fairly rugged, very accurate, and quite expensive. A battery-powered readout unit is not available.

9.3.4 McLeod Gauges

McLeod type gauges are primary type pressure measuring instruments. They have the capability of being quite accurate and are independent of gas composition. Most of the McLeod gauges are made of very fragile glass and certainly couldn't be recommended for field type use at KSC. However, at least one company offers a small rugged mechanically actuated McLeod gauge which is well suited for field use at KSC. The external portion of the gauge is mostly metal. The operation is very quick and simple and there are no electronics involved. In operation, the inlet tube would have to be connected to the vacuum probe space with portable connectors and the probe space vacuum pumped to 5 - 10 microns pressure, and then checked for leaks by monitoring the McLeod gauge. After determining that the connectors are tight and no leaks are present, the main vacuum space bellows type valve would be opened, and the main vacuum spaces could be checked very easily by merely depressing the operating lever and reading the pressure directly. This particular instrument has a maximum pressure limit of 380 torr.

McLeod gauges have one disadvantage that most other vacuum instruments do not have, in that McLeod gauges are sensitive to

condensable gases. In operation, a known volume of gas is trapped and compressed isothermally to a much smaller volume inside a small capillary tube, and the difference in mercury height between the trapped tube and the tube exposed to system pressure is calibrated in terms of pressure. The only condensable gas to be concerned with inside the vacuum spaces at KSC is water vapor. If the trapped gas inside the closed manometer tube contains water vapor, and the water vapor reaches a partial pressure of ≈ 24 torr at 77°F , the water vapor would condense to liquid and an erroneous pressure reading would result. A few calculations will be made to determine the possibility of water condensing inside the closed tube of the McLeod gauge. An average temperature of 77°F will be assumed. Three typical cases will be considered.

Case 1: Pressure inside the vacuum space is 4 torr and the water partial pressure at 80% relative humidity is ≈ 19 torr.

The water partial pressure inside the vacuum space is $\frac{19}{760} \times 4$ torr = $\frac{76}{760} = 0.1$ torr = 100 microns. At 4 torr pressure, the difference in height (Δh) of the mercury column is 32 mm, and the pressure inside the trapped column is $32 + 4$ mm hg or 36 torr. The water partial pressure inside the trapped column is $\frac{19}{760} \times 36$ torr = .9 torr = 900 microns. This partial pressure is considerably less than the 24 torr required for condensation to occur. So, sampling the vacuum space containing air with the same water ratio as outside, at a pressure of 4 torr, would cause no condensation to occur within the McLeod gauge tube.

Case 2: Pressure inside the vacuum space is 150 torr and the water partial pressure inside the vacuum space is $\frac{19}{760} \times 150$ torr = 3.75 torr.

At 150 torr pressure, the Δh of the mercury column is 108 mm, so the pressure inside the trapped column is $108 + 150 = 258$ torr. The water partial pressure is $\frac{19}{760} \times 258 \text{ torr} = 6.4 \text{ torr}$. Again, the water partial pressure is less than the 24 torr required for condensation to occur.

Case 3: Assume a vacuum space containing charcoal sorbent that has just been pumped and that a lot of water was pumped from the charcoal at the lower pressures (10 - 50 microns), leaving the vacuum spaces at 50 microns pressure consisting of 100 % water vapor.

At 50 microns pressure, the Δh of the mercury column is ≈ 18 mm, so the pressure inside the trapped column of the McLeod gauge is ≈ 18 torr. If it is assumed that the gas is all water vapor, this pressure is still below the 24 torr required for condensation to occur. If the ambient temperature decreases to 50°F , the water will condense at a partial pressure of 9.2 torr instead of 24 torr, and in case 3, water would condense inside the closed tube of the McLeod gauge. However, this is an unlikely set of conditions.

In the three cases above, the assumed water vapor content inside the vacuum space is probably higher than would be encountered in actual practice. The vacuum spaces containing molecular sieves would contain much less water vapor.

From the above calculations, it is reasonable to conclude that the chance of obtaining an erroneous pressure reading in the range up to 150 torr, due to water condensing in the McLeod gauge, is not too likely.

Another disadvantage of McLeod gauges is the possibility of contaminating the vacuum system with mercury vapor. This should not be

a problem if the McLeod gauge is used briefly once a month to check the vacuum pressure level, as previously suggested. The mechanically actuated McLeod gauge has the decided advantage of being very simple, rugged, inexpensive, and having a very desirable pressure range, with very good accuracy in the absence of condensible vapors.

9.3.5 Molecular Vacuum Gauges

The molecular vacuum gauge has an operating range of 10^{-3} to 10 torr. One typical gauge consists of two concentric cylinders. One cylinder rotates at 3600 rpm and the other is constrained by a spring and is connected to an indicating pressure dial by appropriate linkage. For a given rotational speed of the moving cylinder, the torque exerted on the constrained cylinder is proportional to the viscosity or pressure of the gas. This type gauge has a desirable pressure range but has many other undesirable characteristics, such as working parts, mechanical linkage, fairly close tolerances, and rotating seals, and is considered too complex to recommend for use at KSC.

9.3.6 Ionization Gauge With a Radioactive Source

One rather unique ionization gauge employs a radioactive source. The gauge contains a small radioactive source which emits low energy alpha and beta particles that ionize the molecules of the gas to be measured. The ions are collected and the charge is amplified with suitable electronics for direct reading on a pressure scale. The radioactive source is a 100 microcurie disc of Radium 226, with a half life of 1620 years, which provides a very stable ionization source through the years. The gauge has a pressure range of 10^{-3} torr to one atmosphere, which is desirable for the KSC application. The gauge is not harmed by exposure to atmospheric pressure because there is no filament to burn out as with some of the other types of ionization gauges. The gauge has a repeatability of $\pm 2\%$ of full scale, with seven different scale ranges,

and is sensitive to the type of gas being measured. The gauges are calibrated with air and there is a correction factor of 4 for hydrogen and 7.7 for helium gas.

Probably, the biggest disadvantage of this type gauge is that the vacuum meter and probe are sold, and must be used, in matching units only, which sell for \approx \$800/matching unit. The vacuum probe for this unit is relatively large (2-1/2-inch diameter x 9-1/2 inches long), and the connection is an open 1-inch O. D. tube. A battery-powered meter is not available for these instruments. Because of these disadvantages, the probe was considered not suitable for use at KSC.

9.3.7 Quartz Bourdon Tube Vacuum Gauge

Another type of vacuum measuring instrument employs a fused quartz, spirally-wound tube to measure pressure. For absolute pressure measurement, the interior of the quartz tube is permanently evacuated and sealed. The system vacuum is opened to the exterior of the tube which is sealed inside a glass capsule. An axial quartz rod is attached to the free end of the spirally-wound tube. The rod has a mirror attached at the bottom. A pressure difference between the interior and exterior of the tube causes the tube and mirror to rotate. Two photo cells rotate in a plane perpendicular to the axis of the quartz rod until the light reflected from the mirror falls equally onto the pair of matched photo cells. The photo cells are mounted on a disc shaped gear. The gear is rotated to the null position by a servometer circuit, and the gear is calibrated in terms of pressure which may be read in digital units.

The quartz tubes are available in various pressure ranges. The lowest pressure range is 0 - 5 psi which gives a readout resolution of 2.65 microns. The hysteresis and fatigue of these instruments is

listed as negligible. These vacuum instruments are very sophisticated, fairly delicate, very expensive, and are deemed not suitable for the field type use incurred at KSC.

9. 3. 8 Ionization-Pirani Gauge Combinations

Several reputable companies have combined the very low pressure measuring ability of the Bayard-Alpert ionization type gauges with the higher pressure measuring abilities of the thermistor type gauges, to make a combination vacuum measuring instrument for the pressure range from 2×10^{-10} to 1000 torr. This type system usually requires two different probes and is much more sophisticated and expensive than what is required at KSC. The lower pressure reading capability of one micron is plenty adequate at KSC, and the two different probes present problems in addition to the added expense. An instrument of this capability is not required for the KSC application.

9. 3. 9 Dial Type Pressure Gauges

Another possibility for making rough pressure measurements is a dial type pressure gauge. One company offers an absolute pressure gauge with a range of 0.1 to 20 torr, with a 6-inch dial. A better suited gauge with a full scale reading of 4 or 10 torr is not available. This gauge will not be damaged by an overpressure of 2 - 3 psi above atmospheric. The minimum graduation on the gauge scale is 0.1 torr or 1000 microns. The precision of the gauge is 0.33% full scale or 66 microns. The price of one gauge is \$240.00. These gauges are fairly delicate, the pressure range is not suitable, and they are not designed for field type work. Because of these disadvantages, this gauge cannot be recommended for use at KSC.

9.4 Other Types of Vacuum Gauges Not Considered

Four other basic types of vacuum gauges were not considered. These are listed below.

- 1) Radiometer or Knudsen gauge
- 2) Thermionic ionization gauge
- 3) Cold cathod ionization gauge
- 4) Bayard-Alpert ionization gauge

The above vacuum instruments were not considered, because the upper limit of their useable pressure range is too low. The Knudsen and Bayard-Alpert gauges have an upper pressure limit of 10^{-3} torr. The thermionic ionization gauge has an upper pressure limit of 10^{-2} torr, and the upper pressure limit of the cold cathode type gauge is 5×10^{-1} torr. None of the above gauges were evaluated because the pressure range was not suitable.

9.5 Gauges Selected for Final Consideration

From the nine different types of gauges evaluated, only four were considered acceptable for final consideration, and these fall into two different groups.

Group I consists of the vacuum instruments with a permanent type probe that would be screwed into the vacuum probe space or directly into the main vacuum space. The readout meters would be battery or ac powered and only a few meters would be used to read all the probes.

Group II consists of the vacuum instruments that have a probe or sensing head which is not well suited for permanent attachment. The sensing probe would not be permanently installed in the transfer line. It is suggested that a gauge from this group of instruments could be used in the following manner.

- 1) It will be assumed that a high-vacuum valve exists between the main vacuum space and the probe space, similar to the existing probe space on the cross-country V. J. lines.
- 2) An O-ring, quick-disconnect, axial-slip-type coupling could be installed in the probe space to facilitate connection of the probe and a small vacuum pump.
- 3) The vacuum measuring probe, readout meter, and vacuum pump would be connected.
- 4) The probe space would be evacuated by a small vacuum pump to ≈ 1 micron pressure.
- 5) After pumping the probe space, the pump would be valved off and a pressure-rise leak check of the vacuum space would be conducted.
- 6) After ascertaining that the probe space has no leaks, the high-vacuum valve into the main vacuum space would be opened, and after attaining equilibrium, the pressure inside the main vacuum space could be read. This procedure would be repeated at each vacuum section.

This type of vacuum measurement has many advantages over the present system of vacuum measurement and should be seriously considered. With the present system, the vacuum probe is subject to corrosion or erosion from salt, sand, and wind all the time, and is subject to vibration and shock during launch. Any leakage into the probe space is dumped into the main vacuum space. One vacuum meter is being used for many different probes, resulting in calibration problems, and the probes are sensitive to gas composition.

Using the newly proposed plan, a matching vacuum probe and readout meter could be used for checking every vacuum space. Non-corrosive caps could be screwed over the slip type couplings, when not checking pressure, instead of the standard caps with holes in them. The vacuum probe would not be subject to weathering or vibration and shock, and would be stored inside most of the time.

The probe spaces would have to be pumped, but this should be done under the current procedure, due to leakage into the probe spaces. Electrical power would have to be provided near the probe boxes to operate a small vacuum pump and the vacuum instrument. A first class, high-quality vacuum instrument system, ideally suited for the entire pressure range, could be used at little additional expense to measure pressure with a high level of confidence. The calibration of the vacuum instrument(s) could be checked periodically under controlled conditions indoors, using a reference gauge such as a good McLeod gauge.

The vacuum measuring instruments considered acceptable which fall into group I are the following:

- 1) Thermocouple or thermopile type gauges
- 2) Pirani or thermistor type gauges.

The thermocouple, thermopile, and thermistor type gauges all offer vacuum probes that may be permanently screwed into the probe space. They all operate on essentially the same principle, and are linear only in the range from 10 to 200 microns, as indicated in figure 9-1. Some of the vacuum meters are calibrated to 5000 microns, and a few brands are calibrated to even higher pressures.

If it is decided that a permanent probe similar to those presently in use is the desired method of vacuum measurement, advantage should

be taken of a recent experimental investigation of various thermocouple and thermopile type vacuum probes by Ametek/Straza (Contract No. NAS 10-6098, Task V). This investigation showed that one manufacturer's product was superior to most other thermocouple or thermopile type vacuum probes for the KSC application.

The probes were extensively evaluated with experimental tests on functional ability, over-pressure, salt fog, sand and dust, temperature extremes, vibration and shock, and accelerated life. Eight probes each, of three different brands, were given the above tests. As a result of the investigation, a primary source and a secondary source of probes were recommended; both were thermocouple type probes.

No thermistor type probes were tested in the above investigation because of some alleged early difficulties with maintaining a good reference vacuum around the reference thermistor. Regardless of the above investigation, it is doubtful that any of the currently available thermistor type probes would perform better under the aforementioned test conditions than the probes recommended. The thermistor sensing element has more mass, which would tend to make this type probe much more sensitive to vibration and shock.

If a group I type vacuum instrument is desired, it is felt that the probe recommended by the Ametek/Straza investigation is definitely the probe to use. There is some question about the sensitivity and accuracy of thermocouple type vacuum gauges in the range of 1 to 5 torr. It is felt that this is pushing the range of this type gauge. If the decision is made to use this type instrument, the accuracy of the meters in this pressure range should be determined.

The gauges selected for final consideration in group II were:

- 1) The capacitance manometer, and
- 2) A mechanically actuated McLeod gauge.

The capacitance manometer is a primary type pressure measuring instrument with a very desirable pressure range of 10^{-5} to 1000 torr. It is a very accurate instrument and is not sensitive to gas composition. The sensing head is quite rugged and the instrument will measure condensible and non-condensable gases. Capacitance manometers are fairly expensive ($\approx \$1500$ for a probe and readout meter); however, if used in the manner suggested, only one or two would be required.

The mechanically actuated McLeod gauge also has many desirable characteristics. The gauge is simple with no electronic circuits. It is fairly rugged and has a very desirable pressure range. One particular gauge does have an upper pressure limit of 380 torr, but this could be easily checked with a compound type dial gauge. There is the possibility of measurement error due to water vapor being condensed in the process of measuring the pressure, but condensation would not occur at pressure levels of concern (less than 150 torr). The mechanically actuated McLeod gauge is not too expensive ($\approx \$200$), and should be considered for use as a calibration type gauge for any other vacuum instrument chosen. It is felt that the thermocouple gauge, capacitance manometer, and mechanically actuated McLeod gauge all have desirable characteristics and should be considered for use at KSC.

The manner in which they could be used also merits some discussion. Two vacuum checking procedures which might be used are explained below.

- (a) The improved thermocouple probes [Amatek/Straza - 1970] and dual range meters may provide the accuracy and reliability that is

desired. If these are used, it is recommended that (1) a good high vacuum valve be installed between each probe and the main vacuum space, (2) the probe space be evacuated before opening the valve, and (3) the meters should be periodically checked and calibrated inside at a central location, with a mechanically actuated McLeod gauge or capacitance manometer. This method of operation would be similar to the present system of vacuum maintenance; it would discontinue the practice of dumping any probe space leakage into the main vacuum space, and it would provide a confident periodic check of the meter calibration.

A second type of vacuum checking procedure (b) would consist of using a vacuum pump-instrument panel system which would be fairly portable and could be easily moved from section to section. The vacuum pump would be used to evacuate the probe space, and the instrument panel would consist of several vacuum instruments to cover all the vacuum ranges desired. The panel might consist of a compound dial gauge and a mechanically actuated McLeod gauge or a compound dial gauge and a capacitance manometer. An axial slip type coupling would be used to connect to the probe space. Such a system would not be expensive, only a few would be required, and a high degree of confidence would result in the vacuum measurements taken. Choosing one of the above procedures will undoubtedly depend on the level of confidence desired in the vacuum measurements.

It is felt that any meaningful experimental evaluation of the above vacuum instruments should be conducted at KSC under the corrosive, field type conditions encountered there, instead of in the controlled conditions of a laboratory.

10.0 Conclusions and Recommendations

Carbon dioxide should not be used for a condensing gas in the transfer line vacuum spaces at KSC. Vacuum spaces must be specifically designed for using the CO_2 condensing-vacuum technique, and the vacuum spaces at KSC were not designed for a condensing-vacuum application. The CO_2 condensing-vacuum technique appears to be marginal when the vacuum spaces are specifically designed for its use, and the technique offers no advantage if small leaks are present. The present state of the art for high-vacuum transfer lines far exceeds that of transfer lines using the condensing-vacuum technique.

Coconut charcoal is preferred and recommended over molecular sieves for use as a sorbent in non-LOX transfer line vacuum spaces, especially if the transfer line is to be used in a high humidity environment such as at KSC. Charcoal is preferable, mainly because water may be desorbed at ambient temperatures. The sorption characteristics of charcoal for hydrogen are better than molecular sieves and are as good, if not better, for the atmospheric gases (air). However, charcoal is not LOX-compatible and is not recommended for use in the vacuum spaces of LOX transfer lines.

Vacuum spaces containing charcoal sorbent should be pumped for a sufficiently long period of time to desorb water and attain a steady vacuum space pressure of 10 microns or less after pumping has been stopped. Water is easily desorbed from charcoal at ambient temperatures and pressures below 2 - 3 torr, but the desorption process may require a long pumping period.

Molecular sieves have a very strong attraction for water at very low partial pressures of water. This characteristic is very desirable in some applications, but is a decided disadvantage in a cryosorption application in a high humidity environment. It is a disadvantage because

at 2 weight percent presorbed water, the sorption capacity of the sieves decreases, and above 7 weight percent, the sorption capacity is almost completely destroyed. Because of this characteristic, special precaution should be exercised when making repairs to vacuum spaces containing molecular sieve sorbent. It is most important to keep the atmospheric moisture out of the vacuum space. This might be accomplished by purging the vacuum space with nitrogen gas while making repairs at one end. Vacuum spaces containing molecular sieves should never be allowed to self pressurize with atmospheric air and moisture. The spaces should be pressurized with dry nitrogen gas.

If there is reason to believe that the molecular sieves in a vacuum section have sorbed a significant amount of water, the vacuum space should be given a vacuum-bakeout at a minimum temperature of 150 - 200°C. A vacuum-bakeout at the recommended activation temperature of 350°C and 10 microns pressure is even more preferable. It is most important that the water content of the molecular sieves be less than ≈ 2 weight percent to insure that the sieves have their rated sorption capacity.

If there is reason to believe that helium is present in a vacuum space due to leak detection operations, the vacuum space should be repeatedly pressurized with nitrogen and vacuum pumped until most of the helium has been pumped out of the interstitial spaces of the multi-layer insulation. It is most important to remove the helium from the vacuum spaces of cryogenic transfer lines, and especially those operating at 90 K. Helium is very weakly sorbed at 90 K and is not too strongly sorbed at 20 K.

An analysis of the heat leak to the liquid oxygen and liquid hydrogen systems reveals that the heat leak due to residual gas conduction

within the vacuum spaces is a very important parameter. At 10^{-4} torr pressure inside the vacuum spaces, the heat leak due to residual gas conduction is only 1% of the total system heat leak. At 10 microns pressure within the vacuum spaces, the total system heat leak increases by 10 and 24%, respectively, for the hydrogen and oxygen systems, and at 100 microns pressure, the total system heat leak increases by 64 and 286%, respectively. So, very significant increases in system heat leak can result from excessive pressures within the vacuum spaces.

After evaluating the cryosorption characteristics of the two sorbents, along with other pertinent parameters, it has been determined that a 4000 micron ambient pumpout pressure level would be satisfactory for the vacuum spaces of the hydrogen transfer lines. Calculations indicate that either of the sorbents should easily cryosorb the gas to an insulating vacuum of 10^{-4} torr, provided the sorbents are properly activated. However, the cryosorption characteristics of the 90 K liquid oxygen transfer lines are considerably different from the 20 K hydrogen lines. After evaluating the pertinent cryosorption parameters, it appears as if an upper pressure limit of 1000 microns is preferable for the liquid oxygen vacuum spaces.

Fresh capsules of palladium monoxide should be inserted and broken inside the LOX transfer line vacuum spaces periodically (≈ 1.2 grams/year). Hydrogen is very weakly cryosorbed at 90 K and must be converted to water, which is strongly sorbed by the molecular sieves. Adding fresh capsules of palladium monoxide to the liquid hydrogen vacuum spaces is not as important, provided the sorbent is activated. Hydrogen gas is easily cryosorbed at 20 K.

The dresser-type joint covers on the LC-39A transfer lines at KSC should be replaced with permanent sealed covers (possibly welded

or sealed with good O-ring seals) and be evacuated through a good pumpout valve. This would improve the operation in several ways. It would decrease the heat leak to the joint section, and it would also help solve the recurring problem of the annular end seal failures, by providing a vacuum on both sides of the annular end seals.

A survey of other cryogenic facilities using large vacuum insulated transfer lines revealed that most other installations are using thermocouple type vacuum gauges with reasonable success. However, most installations do not have the corrosive environment like that at KSC. Several of the facilities had a pumpout level of 1000 microns, but none had a pumpout level above 1000 microns.

Various types of vacuum gauges were evaluated for possible use at KSC and three were chosen to be recommended for consideration. The vacuum gauges recommended are:

- 1) A thermocouple probe with matching dual scale meter to 5000 microns (Amatek/Straza, 1970), and
- 2) A capacitance manometer, and
- 3) A mechanically actuated McLeod gauge.

A different procedure should be adopted for checking the pressure in the vacuum spaces. Atmospheric air that has leaked inside the vacuum probe space should not be dumped into the main transfer line vacuum space. This practice admits air and moisture into the main vacuum space and may significantly shorten the pumpout interval. The probe space should be evacuated with a small vacuum pump before checking the pressure inside the main vacuum space. Procedures (a) or (b), as outlined in section 9, are recommended for consideration. It is felt that either of the procedures would give much better confidence in the vacuum measurements, especially procedure (b) employing a complete vacuum pump-instrument panel system.

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COOLDOWN PRESSURE SURGES

(Part II)

Roland O. Voth

1.0 INTRODUCTION

A study has been conducted of the pressure and flow surges generated during cooldown of the 14-inch cross-country liquid oxygen line located on LC-39 at the Kennedy Space Center. The purpose of the study was to determine if the pressure surges currently being measured during cooldown could be predicted using a simplified mathematical model and to determine from the model if any damaging pressure surges may be present at other points along the line. Also, if the model successfully predicts the current pressure surges, changes in the cooldown procedure could be investigated using the mathematical model, saving the expense of experimental determination of pressure surges. The study was conducted to fulfill the requirements of Phase I, NASA Contract No. CC-59384.

Measured pressure surges tend to exhibit large scatter because of the many variables influencing the surges. Even during well controlled experiments, variations in the generated peak pressures have been found. Thus, a mathematical model to predict the magnitude of pressure surges becomes very complex; however, a simplified model developed by Steward [1964] was used successfully to predict the initial pressure surge, within the experimental scatter, during cooldown of a 200-foot long by 0.625-inch I. D. transfer line with liquid nitrogen. This mathematical model was applied to the Kennedy system and an initial pressure and flow surge calculated for the system.

The calculations showed that the current cooldown procedure plus the mechanical complexity of the Kennedy system tended to minimize the magnitude of cooldown pressure surges. Two important parameters

which can cause high cooldown pressure surges are the amount of sub-cooling present in the initial inflow of liquid and the quantity of the initial inflow of liquid. The current cooldown procedure has an initial inflow of saturated liquid because the uninsulated line between the inlet valve and storage vessel is filled with liquid prior to initiation of cooldown. The quantity of liquid involved during the initial inflow is limited by a slow-opening inlet valve and a filter and pump located a short distance downstream of the inlet valve. The location of the pump and/or filter, however, does produce a water hammer type of pressure spike which apparently has not caused damage to any components in the system.

Preliminary calculations of cooldown time for the Kennedy system have also been performed. During these calculations, a mathematical model was developed that includes the effect of heat transfer to an uninsulated line during the cooldown period. Since the heat transfer rate to an uninsulated line depends on weather conditions and time the line has been cold, an average heat transfer rate for the cooldown period is difficult to determine. However, the effects of the heat transfer rate, the pressure ratio maintained during cooldown, and the vent size are demonstrated (based on the model).

2.0 WORK BY OTHERS

Much of the information available from the literature concerns simplified treatments of the cooldown problem. The primary emphasis of most studies is the calculation of cooldown time, with very few studies concerning the pressure surges generated during the initial phases of cooldown.

Burke [1960] and Drake [1961] present a method to calculate cooldown time. They used a "cold front" type of process; that is, a steadily advancing cold front was assumed in the pipeline. Upstream of the front, the pipe was assumed completely cooled, and downstream,

the pipe remained at its starting temperature with the discharge gas at an average temperature. The model neglected the possibility of pressure and flow oscillations during the cooldown process.

Another cooldown approach using a moving "cold front" process is presented by Steward [1968]. His model integrates the flow in a generalized form over the length of the pipeline and requires the averaging of only the friction factor. Macinko [1960] used a cooldown model in which the pipe was assumed to cool uniformly over its length. The boil-off gas temperature was also uniform along the length and was assumed to be 10°C colder than the pipe wall (a value which produced the best fit of the experimental data). Neither of these two models allowed or calculated any pressure or flow oscillations during the cooldown period.

Bronson [1962] observed a "cold front" type of cooldown in his transfer system used for supplying liquid hydrogen to nuclear rocket engines. The main concern of the study was the possibility of stratified flow in 8 and 10-inch diameter pipes. The stratified flow of liquid running along the bottom of the pipe with relatively warm gas above could cause bowing of the lines because of unequal temperatures around the pipe circumference. Bronson observed a "cold front" type of cooldown, and he also derived a method to predict the frequency of pressure oscillation during cooldown. The method was only partially successful, predicting the frequency but not the amplitude of the pressure oscillations.

Thurston [1966] studies an instability phenomenon, not associated with cooldown, termed "thermal-acoustic" oscillations for liquid hydrogen. These oscillations occur in otherwise steady flow under special conditions of pressure, heating, and flow which produce a dense core of fluid surrounded by a superheated vapor film near the inlet. Acoustic frequencies of Helmholtz and open pipe resonance were used as a

reference for correlation of the observed frequencies, and "boiling numbers" were used to correlate the amplitude of the pressure oscillations.

There have been several attempts at predicting complete cool-down history by solving the conservation equations in various degrees of simplification. Jarvis [1965] tried a model permitting both thermal and mechanical nonequilibrium by allowing separate temperature distributions for both the liquid and gas as well as separate velocities. Jarvis used the Eulerian reference system, but suggested that the Lagrangean approach might have some natural advantages for a one-dimensional system.

Aerojet General Corporation [1964] used the Eulerian system of reference. An array of fluid and solid "nodes" was distributed throughout the complex system of a nuclear rocket's coolant lines, pump, reactor core, and nozzle. Two modes of operation were possible: (1) with a known inlet flow rate, the resulting system transients could be calculated; (2) with an inlet pump of known pressure-flow characteristics, the resulting system transients are calculated.

Koshar [1960] predicted loading time, gas venting rates, and temperatures for missile oxidizer pipelines. The computation was able to include complex piping systems; however, it did not deal with pressure and flow surges since the liquid and gas were treated as incompressible. Even in the absence of surging, the computation was very complex and required compounded iterations to arrive at the correct flow distribution in various branches.

Other references which attack the cooldown problem are listed in appendix III. Most studies only measured cooldown time or pressure surges in a particular system and do not attempt to model the measurements mathematically.

Other general references used in this study include: the oxygen properties - Weber [1971], heat transfer information - Brentari [1965], and sonic velocity in two-phase flow - Smith [1963].

3.0 DEFINITION OF CURRENT COOLDOWN PROCEDURES AND IMPORTANT SYSTEM VARIABLES

To effectively model the system, the current cooldown procedure as well as the important mechanical features effecting the pressure and flow surges had to be defined. Table 3.1 shows the sources of information used to define the system.

Table 3.1

Specifications and Drawings Used to Define the KSC System

Number	Title
76K04827	Saturn V Liquid Oxygen Loading Criteria LC-39A
75M21704	LC-39 ML LOX System
75M05898	Complex 39A LOX System Mechanical
75M21466	Complex 39 LOX System Mechanical Umbilical Tower Piping Details
75M05869	Specifications, Complex 39A LOX System Mechanical
TM-437-D	Maintenance Instructions Liquid Oxygen System Complex 39, Launch Area A

The cooldown procedure is important to the calculation of pressure surges for approximately 5 minutes after the introduction of a flow transient in the system. Since during the present cooldown procedure, the only transient introduced is the opening of the inlet valve (A1), only the initial portion of the procedure is critical. System

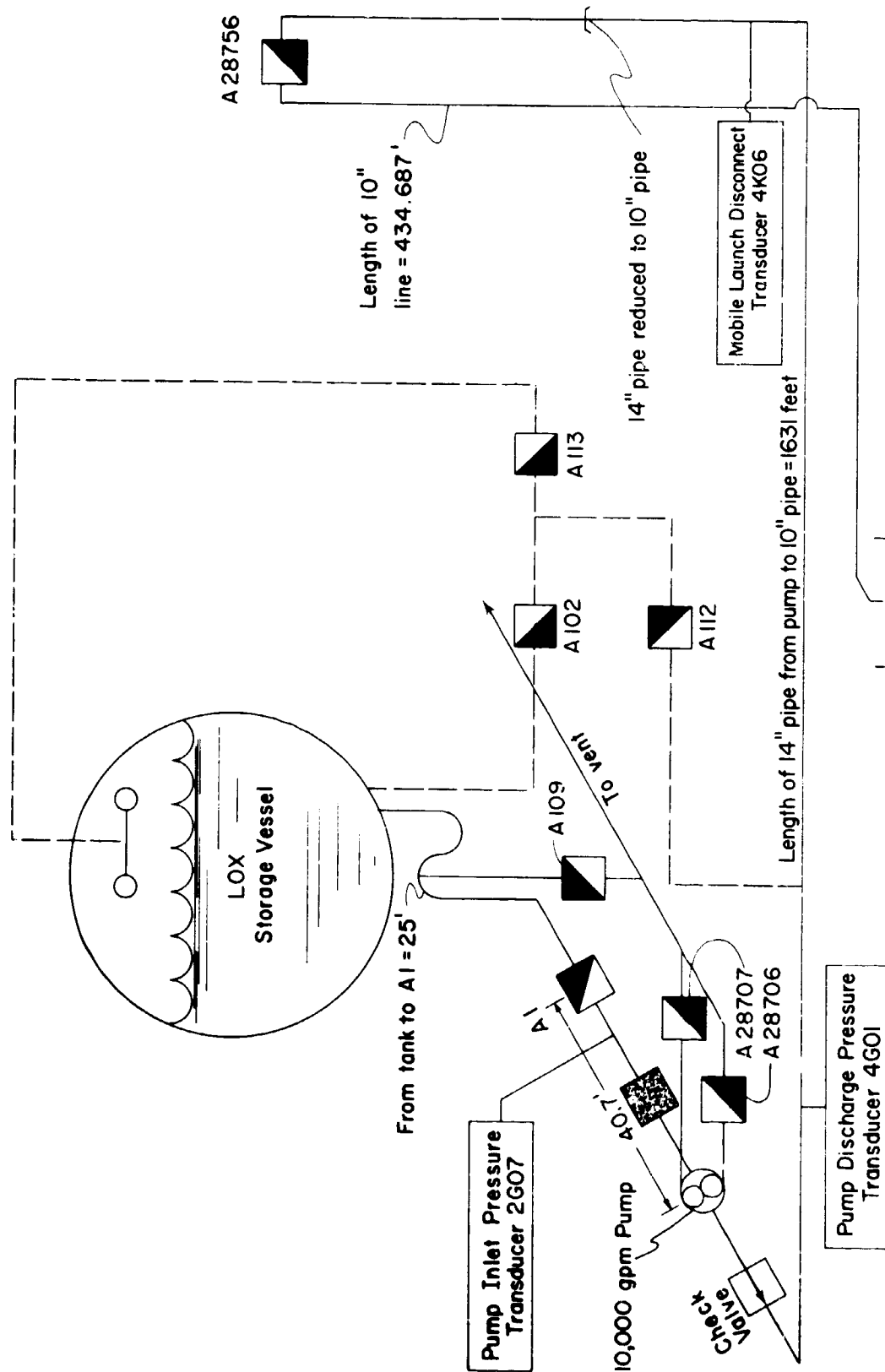


Figure 3.1 Schematic of 14-Inch Cross Country Liquid Oxygen Line

mechanical features are shown in figure 3.1. Cooldown is initiated by opening valve A109. This allows the line between the storage tank and valve A1 to be chilled and filled with liquid. After an extended period, cooldown of the main system is initiated by opening valves A28756, A28706, A28707, and finally valve A1. Valves A28706 and A28707 are closed approximately 1 - 3 minutes later; otherwise, the configuration remains unchanged for at least 15 minutes. During these initial stages of cooldown, the 10,000 gpm pump motor is started, but the magnetic clutch between the motor and pump is not engaged, allowing the pump to turn freely. Calculations of cooldown pressure surges were made using the time when valve A1 was opened as time zero.

Physical parameters important to the mathematical model are the opening characteristics of valve A1, the length of line between the tank and the inlet valve (X VALVE), the temperature of the liquid admitted during the initial inflow, and the downstream restriction to the flow of gas generated during the inflow.

The opening characteristics of the inlet valve A1, where opening characteristics determine the pressure drop across the valve versus flow and time, are quite important when determining pressure surges - Brennan [1966]. Even though the type and manufacturer of the butterfly valve are known, the exact opening characteristics are not known. The opening time for the butterfly valve (A1) was specified as 5 ± 2 seconds, with 5 seconds being used in the computer program. Information from the manufacturer showed that the valve is actuated with a pneumatic actuator through a lever attachment with a variable mechanical advantage. Since the butterfly opening torque also varies with the degree of opening, actual opening characteristics are difficult to determine. Other information obtained from the manufacturer is shown in figures 3.2 and 3.3. Figure 3.2 shows percent of full flow versus percent

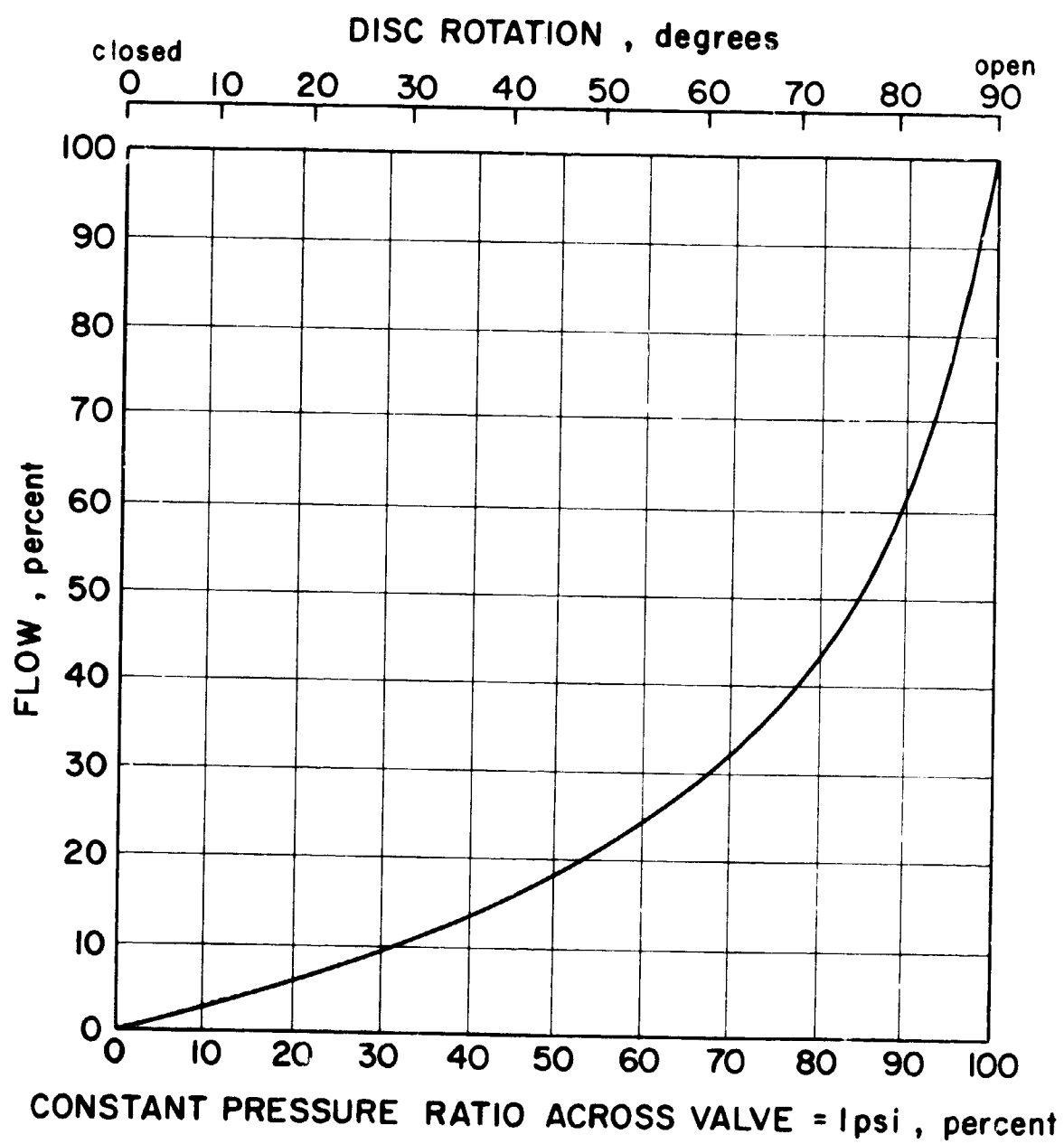


Figure 3.2 Flow Characteristics of the Inlet Butterfly Valve.

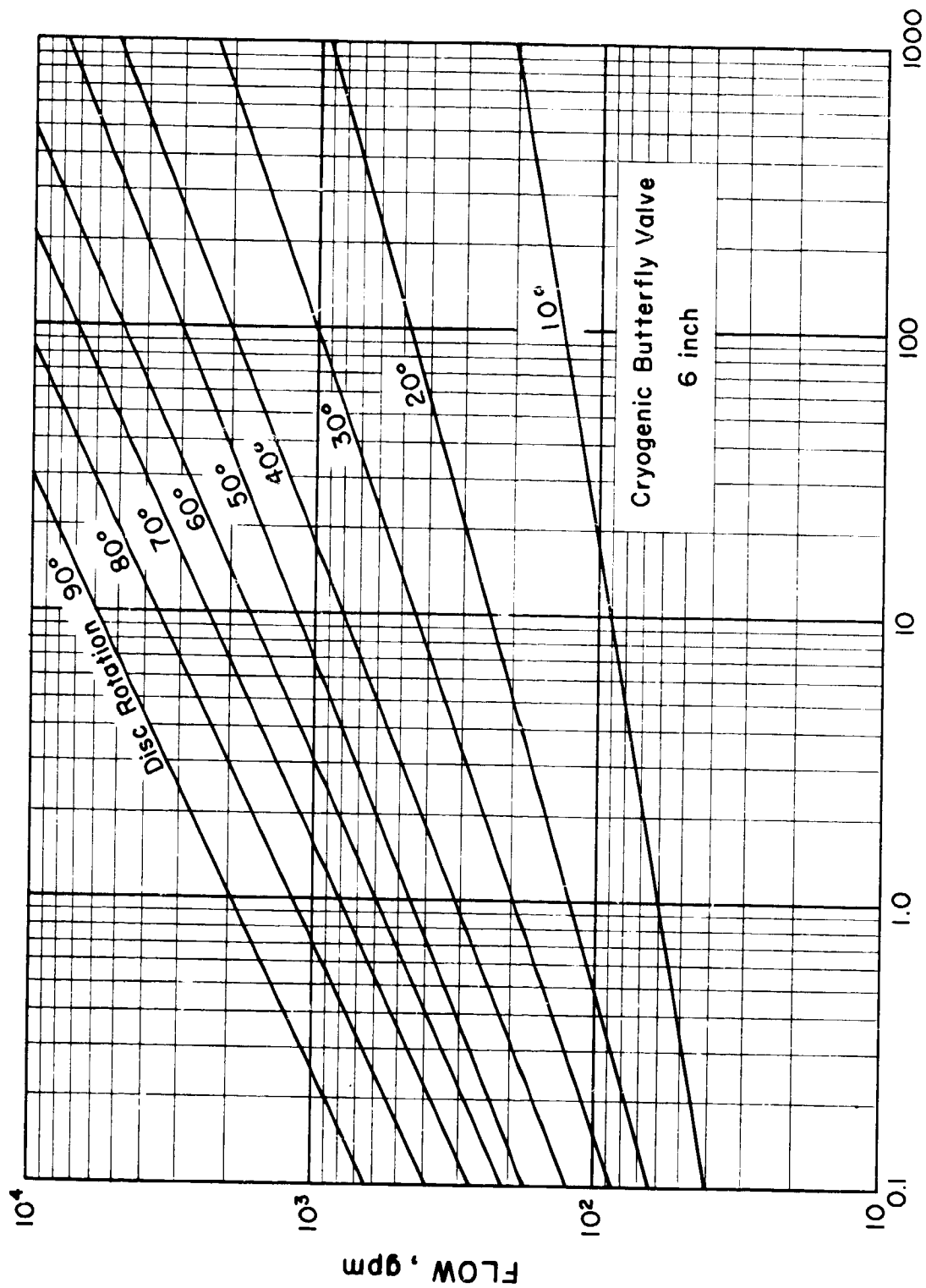


Figure 3.3 Flow Characteristics of a 6-Inch Butterfly Valve.

open at a constant pressure differential across the valve of 1 psid. Figure 3.3 shows the variation in pressure drop versus flow for various openings. The characteristics shown in the two curves were for a 6-inch valve and were determined by the manufacturer using water as the test fluid.

Inlet valve characteristics used in the calculations were derived from these figures. Equation (3.1), following, was fit to the curve shown in figure 3.2 and was used to determine the fractional flow (y) for the valve versus the percent open (X) at a constant pressure drop of 1 psid.

$$y = 1.0 + 2.12 \times 10^{-3} X - 2.12 \times 10^{-5} X^2 - (1.0 - 10^{-4} X)^{-2} \quad (3.1)$$

The fractional flow (y) was multiplied by the total flow for an 18-inch valve 100 percent open with a 1 psid pressure differential. The resulting flow was the fractional flow (Q_F) through the valve at y percent open with a pressure drop of 1 psid. Then the pressure drop was altered from the nominal 1 psid to the true pressure drop by multiplying it by a ratio of flows--raised to a power. The power (exponent) was obtained from the slope of the 20 degree open curve on figure 3.3. The resulting equation was:

$$\Delta P_a = 1.067 \left(\frac{Q_a}{Q_F} \right)^{3.46} \quad (3.2)$$

where ΔP_a = actual pressure differential,

Q_a = actual flow through the valve as determined by the computer,

and

Q_F = fractional flow through the 18-inch valve at 1.067 psid.

The 1.067 psid was obtained by correcting the 1 psid water data to liquid oxygen.

Several opening rates were tried in the mathematical model. The opening rate that fit the measured data best was a linear rate represented by the equation, $X = 20.0 \times \text{TIME}$. Thus, the valve was completely closed at time zero and 100 percent open in 5 seconds.

In the mathematical model, the length of line between the tank and the inlet valve was taken to be the actual length of line. This was 25 feet for the application of the model to the section of line between valve A1 and the pump, and 65 feet for the section of line downstream of the check valve.

The temperature of the liquid admitted during the initial inflow was taken as saturation temperature at the inlet pressure. The inlet pressure was 35 psig and consists of the 11 psig ullage pressure plus the head of liquid oxygen in the tank. The inlet temperature could be taken as saturation because the uninsulated inlet line was precooled and filled with liquid oxygen for a period of time before cooldown was initiated; the liquid in the tank was near saturation at one atmosphere and would be subcooled at 35 psig.

The restriction to flow of gas generated during the initial inflow was obtained by determining an effective length for the piping system. The effective length was the actual length of downstream pipe, plus added equivalent lengths for other components in the system causing pressure drop. Table 3.2 lists the actual length of line and the equivalent lengths of line added for the components.

Table 3.2

Actual and Equivalent Lengths for the KSC System

	Description	Length
1.	Actual length of 18-inch pipe from storage vessel to inlet valve A1.	25.0 feet
2.	Actual length of 18-inch O. D. pipe from inlet valve A1 to pump.	40.7 feet
	Equivalent lengths to add pressure drop for valve A100, filter A103, 1 elbow.	134.55 feet
	Total equivalent length for this section.	200.25 feet
3.	Actual length of 14-inch O. D. pipe from pump to 10.02-inch I. D. pipe.	1631.4 feet
	Equivalent lengths to add pressure drop for 4 - 90 degree elbows, 4 - 45 degree elbows, 1 check valve, 3 tees, flowmeter LFM-2.	392.2 feet
	Total equivalent length for this section.	2023.6 feet
4.	Length of remaining 10.02-inch I. D. pipe to catch basin.	588.3 feet
	Equivalent lengths to add pressure drop for 9 elbows, 3 butterfly valves, 1 check valve, 1 gate valve.	324.0 feet
	Total equivalent length for this section.	912.3 feet

4.0 DESCRIPTION OF THE MATHEMATICAL MODEL

Steward [1964] presents a detailed description of the model used to calculate pressure surges in the Kennedy system. Therefore, a detailed description will not be presented here; however, a description showing the qualitative effect of various physical parameters on the magnitude of the pressure surges and an overview of the assumptions made in the calculations will be presented.

Pressure and flow oscillations will be generated when a cryogenic liquid is suddenly admitted to a warm line or when a sudden change in liquid inflow is made during the cooldown period. Upon opening of the inlet valve to a warm line, the liquid cryogen rushes into the warm line. The liquid boils or flashes, producing vapor at a rate greater than it can escape, creating back pressure, slowing and finally reversing the incoming liquid. Even after the liquid flow is reversed, liquid is continually carried downstream by the escaping gas and brought into contact with the warm line, thus generating additional gas and creating even higher pressures. Finally, the pressure decreases after part of the initial charge has evaporated or has been forced back out of the line. This decrease in pressure again allows liquid to flow into the line and the process repeats. The process continues to cycle with diminishing amplitude until a quasi-steady state is reached or until the line is completely cooled. High initial pressure surges are caused by: (1) using subcooled liquid for the initial inflow, thereby delaying the generation of vapor, and (2) using a quick opening valve at the inlet of the line again allowing large quantities of liquid into the line before sufficient quantities of vapor are generated to slow or reverse the liquid inflow. Other parameters having a minor effect on the magnitude of the pressure and flow surges are the length of line between the tank and the inlet valve, and the length of line downstream of the inlet valve which has been precooled by conduction along the wall of the pipe. Because the initial cooldown process is not in equilibrium, it is difficult to analyze; however, a relatively simple fluid flow model has been successful--predicting the amplitude, location, and elapsed time for the initial peak pressure surge.

The following assumptions were made to allow the derivation of the simplified model:

1. Since the initial pressure surge is the most severe, only the initial pressure surge is calculated by the model.
2. The wall temperature does not change during the initial surge cycle.
3. Since during the cooldown experiments cold fluid did not reach the discharge end of the pipe until near the end of the cooldown period, the approximate model is simplified by assuming that all of the heat transfer, rather than being distributed down the total length of pipe, takes place only at the gas-liquid interface. Additionally, it is assumed that there is a discontinuity in the density, velocity, temperature, and pressure at the interface, and that the vapor stream is adiabatic. These assumptions limit the model to relatively long pipelines.
4. Because of the interface discontinuity, it was necessary to develop a separate heat transfer model. This model supplies a heat transfer area which is simply taken to be the area of warm pipe in contact with the liquid, plus an added area due to the liquid being carried downstream with the vapor.
5. The transfer line is of constant cross sectional area, horizontal, and straight. No restrictions occur along the line except the inlet valve.
6. A single boiling heat flux is considered to be a parameter adjusted for different fluids and pipeline diameters.
7. Quasi-steady-state Fanno flow is assumed for the vapor.

The computer program analyzes the initial pressure surge in small intervals of time. During each time interval, the average pressure acting on the liquid is assumed; the liquid acceleration and a new liquid velocity calculated. From an analysis of the heat transfer, the

changes in pressure, density, and velocity across the interface are calculated. Then the pressure drop due to the Fanno flow with possible choking at the discharge is calculated for the vapor stream. Finally, the calculated pressure on the liquid is compared with the assumed pressure. If the two pressures do not agree, the assumed pressure is adjusted and the calculation repeated. When the iteration calculates a liquid pressure equal to the assumed pressure, the computer proceeds to the next time step.

Application of the simplified model to the complex Kennedy system required the derivation of an equivalent piping system allowing for the various assumptions present in the model. Because the model did not have the capability of including the effects of a check valve located some distance downstream from the inlet valve, two equivalent systems were defined and used to calculate pressure surges in the system. The first equivalent system was used for the line from the inlet valve (A1) to the pump, and the second was used downstream of the check valve, including the remaining portion of the line. Using the two models, the pressure surges for the entire system could be determined.

Another difficulty with the simplified model was the assumption of an equal pipe size for the entire system. Since the vapor pressure drop is calculated from the downstream pipeline configuration, an equivalent constant diameter pipeline was defined giving the same pressure drop as the actual system at identical flow rates. The diameter was chosen to be constant and equal to the smallest diameter pipe present in the system.

To determine the equivalent length of a constant diameter pipe line, the pressure drop equation, $\Delta P = f \frac{L}{D} \frac{\rho U^2}{2g_c}$, can be used. The

variables used in the equation are defined as: ΔP is pressure drop, f is an average friction factor related to a Reynolds number, L is length, ρ is the density of the flowing fluid, U is the velocity of the flowing fluid, D is the diameter of the pipe line, and g_c is a conversion factor. Since the pressure drop through the smaller equivalent pipe must be the same as the larger pipe, the following is written:

$$\Delta P_L = \Delta P_S = \frac{fL_S}{D_S} = \frac{\rho U_S}{2g_c} = \frac{fL_L \rho U_L}{D_L 2g_c}, \quad (4.1)$$

where the subscript L refers to the larger, and S refers to the smaller pipeline. If an equal friction factor and density is assumed for the two systems, the equation becomes $L_S/L_L = D_S^5 U_L^2 / D_L^5 U_S^2$. Substituting $Q = AU$, where Q is the volume flow rate and A is the flow cross section area of the pipe lines $\left(\frac{\pi}{4} D^2\right)$, into the above equation results in $L_S = L_L (D_S/D_L)^5$. Using this equation to determine an equivalent length of smaller pipe from the lengths shown in table 3.2 gives,

$$L_{eq} = 200.25 (10.02/16.875)^5 + 2023.6 (10.02/13.)^5 + 912.3,$$

or $L_{eq} = 1477.5$ ft.

An equivalent length of 1477.5 feet of 10.02-inch diameter pipe was used as input to the computer program.

The constant wall temperature assumed by the model is usually sufficient when the initial pressure surge occurs a short time after the pipe is in contact with liquid. However, for the portion of line after the check valve, the initial peak pressure surge occurred more than 2 minutes after the initial inflow of liquid. Substantial cooling of the wall could occur in this period of time, perhaps reducing the amount of heat transfer. During the computation for this portion of the system,

the heat transfer coefficient was reduced to obtain a good fit of the measured data. Another reason for a reduction in the heat transfer coefficient is the large pipe diameter of the Kennedy system. In the smaller system, originally used to correlate the model, the fluid was probably in contact with the complete circumference of the pipe, while in the larger system, it is probable that the more dense liquid tends to lie in the lower part of the pipe with the gas flowing above the liquid. With the liquid in the lower portion of the pipe, the heat transfer area is reduced. This reduction seems even more reasonable if the inflow of liquid is restricted as is apparent from the calculations and must be due to the freewheeling pump acting as a restriction.

The Kennedy system also includes a riser near the end of the line. This vertical section of pipe was judged not to interfere with the calculations because the liquid does not reach the vertical section during the first surge, and little additional pressure drop is present due to gas flowing up the vertical section. Drake, et al. [1961], measured the cooldown time for a system which contained a vertical downcomer and found no significant effect from the downcomer on either the cooldown time or the oscillations produced during cooldown.

5.0 RESULTS AND DISCUSSION OF THE PRESSURE SURGE CALCULATIONS

The results obtained from the application of the mathematical model to the Kennedy system are presented in two parts. The first part provides results for the line downstream of the check valve and the second part describes the results for the section of line from the inlet valve to the pump. The parts were treated separately because the mathematical model was not capable of providing results for a complex system with a check valve located at an intermediate point. Interaction between the two parts is probable; however, the interaction would be minimal because during any reverse flow, when

highest pressures are generated, the check valve closes, essentially separating the two sections.

A complete listing of the computer program for the portion of the system between valve A1 and the pump is shown in appendix I. A similar program with a restraint not allowing reverse flow was used for the section of pipe downstream of the check valve. Appendix II gives definitions of the input parameters to the computer program. Table 5.1 is a listing of the input values used in the calculation of the pressure surges for both parts of the system.

5.1 Pressures Downstream of the Check Valve

The calculated pressures for the section of line downstream of the check valve are compared with measured pressures in figure 5.1. The measured pressure is a band showing the scatter in measured data for several cooldowns. The measured pressures were obtained from two transducers. Pressures for times from 0 to 180 seconds are from the 10,000 gpm pump discharge transducer (4G 01), and pressures for times after 180 seconds are from the launch umbilical tower disconnect transducer (4K 06). Insignificant differences were noted between the recorded pressures at the two locations. The computer program successfully predicted the maximum pressure and the time when the peak pressure developed. Two areas of minor disagreement appear on the curve: First, the calculated pressure surge has a longer duration than the measured surge, and second, initially the measured pressure increases more rapidly than the computed pressures.

Table 5.1
Values Used as Input to the Computer Program

Variable	Piping Downstream of the Check Valve	Piping Between Inlet Valve A1 and the Pump
DELTX	5.0 cm	1.0 cm
TON	55 s	5 s
XCOLD	0.3 cm	1.0 cm
X4	47035.9 cm	45785.9 cm
DIAM	25.45 cm	25.45 cm
QFACT	4.00 cm	4.00 cm
PIN	3.425×10^6 dyn/cm ²	3.425×10^6 dyn/cm ²
PAMB	1.013×10^6 dyn/cm ²	1.013×10^6 dyn/cm ²
C5	6.3×10^5	6.3×10^5
C6	1.5844×10^{-4}	1.5844×10^{-4}
XVALVE	2000.0 cm	750.0 cm
DENSL	1.0723 g/cm ³	1.0723 g/cm ³
A	0.2	0.2
B	0.2	0.2
C1	17954.0	17954.0
C2	0.0211	0.0211
C3	0.0550	0.1605
C8	2.0×10^4	2.0×10^4
C9	0.0250	0.0250
C10	0.10	0.10
C12	2.055×10^6	2.055×10^6
C13	1.0949×10^{-7}	1.0949×10^{-7}
VSOUND	3.3×10^4 cm/s	3.3×10^4 cm/s
IPRINT	0	0
ISTOP	240	195

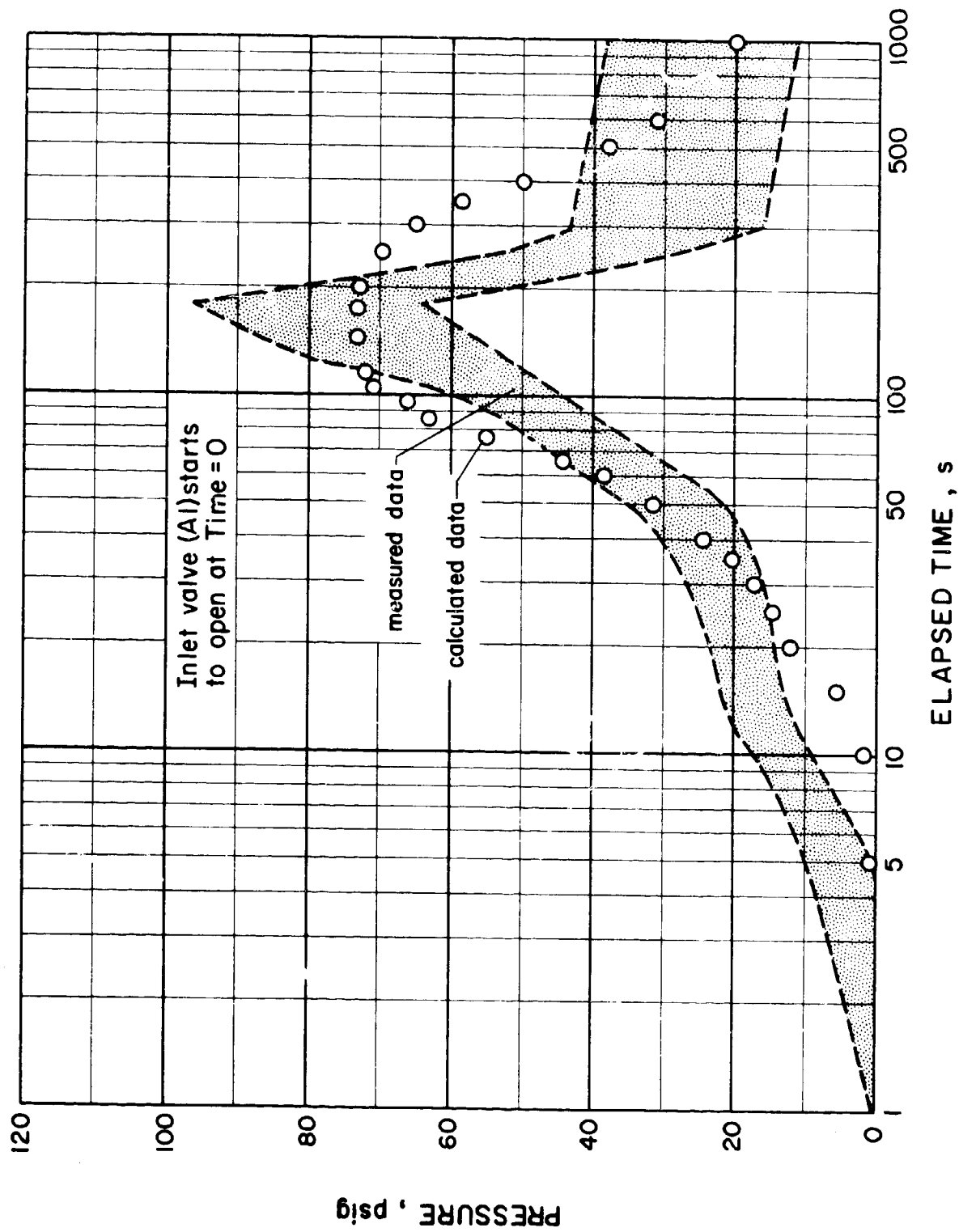


Figure 5.1 Peak Pressure for Line Downstream of Check Valve.

The differences between the calculated and measured pressures are probably due to simplifying assumptions made in the mathematical model. One assumption is that the pipe remains at a constant ambient temperature during the entire pressure surge. This assumption is reasonable when the initial surge occurs early; however, the assumption may no longer be valid for the Kennedy system because of the long time period of the initial surge. If sections of the pipe are cooled to liquid temperature, less gas may be generated, resulting in lower pressures.

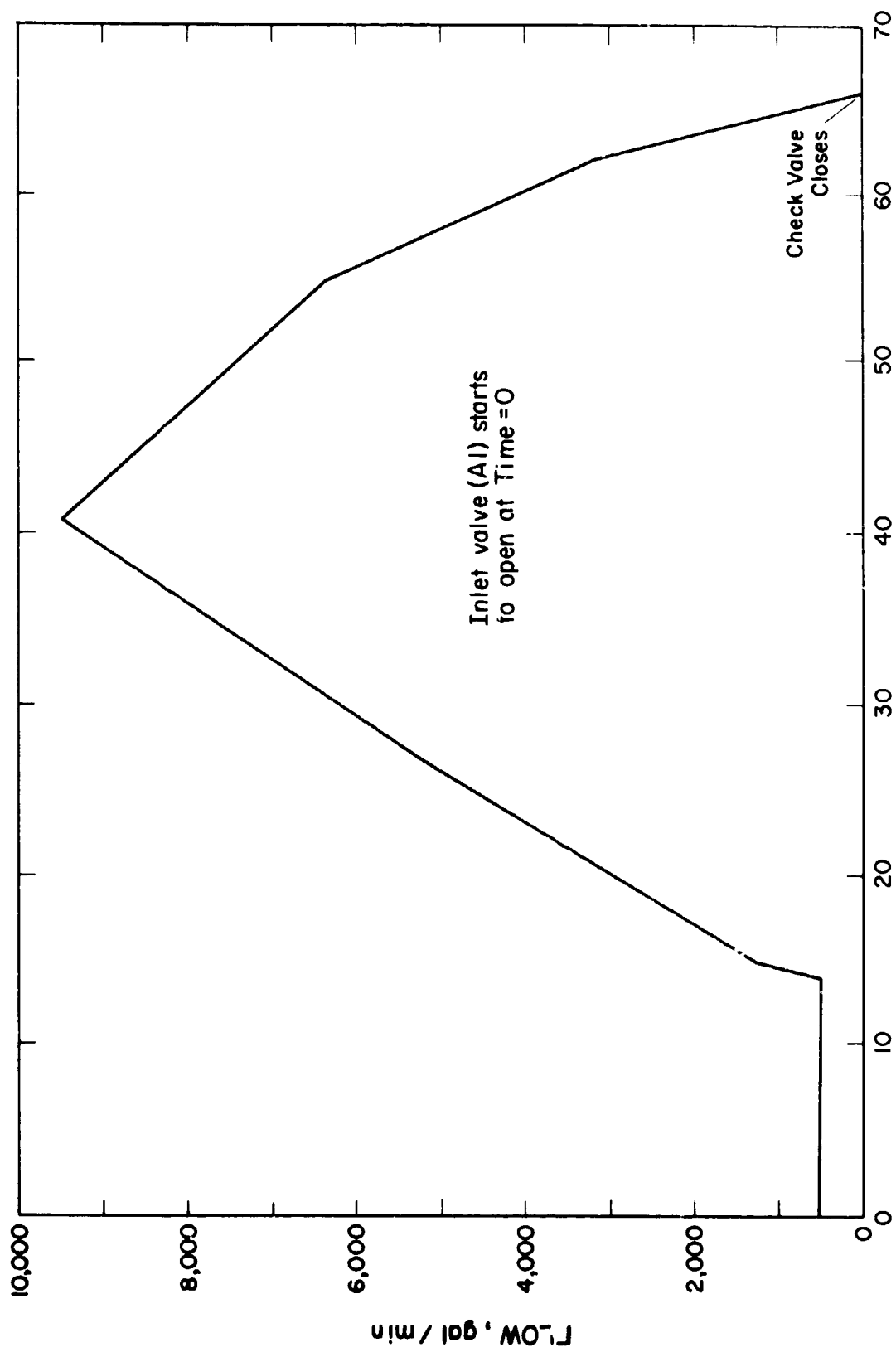
The lower initial pressure rise rate predicted by the computer program may be due to the limitation that only a single phase fluid can be admitted initially during the initial liquid inflow in the computer program. In the actual case, a two-phase fluid or even all gas may flow into this section of line during part of the initial inflow. Whatever the reasons for the slight discrepancies, the calculated results are sufficiently accurate to test different cooldown procedures in the same physical system.

The location of the maximum pressure in the line downstream of the check valve is at the liquid gas interface. The computer results indicate this location to be 121 feet downstream of the check valve. However, since no reverse flow of the liquid is allowed by the check valve and since the pressure surge develops over an extended time, the pressure throughout the liquid portion of the flow will be essentially equal to the interface pressure. The pressure downstream of the maximum pressure point will be reduced by the pressure drop in the flowing gas stream. The major portion of the pressure drop will occur in the 10-inch vent system; therefore, the pressure will drop only slightly in the 14-inch line. Comparing the pressure measurements from two widely separate sensors at the pump discharge and at the launch umbilical tower disconnect with the calculated pressures is, therefore, feasible.

The calculated and measured pressures are not high enough in any location to damage a system designed for a 300 psig working pressure.

Since a freewheeling pump is located between the liquid oxygen vessel and the check valve, a flow characteristic simulating the pump was necessary to obtain accurate results from the computer. Pump flow characteristics, during freewheeling operation, were not known, so the characteristics were varied until a good fit of the measured pressures was obtained. The final flow characteristics chosen were identical to the already defined inlet valve (A1). Only the opening rate was changed to yield a calculated pressure surge comparable to the measured surge. The valve was opened a constant 4 percent for the first 15 seconds of the cooldown period and then opened linearly to 100 percent in an additional 40 seconds. The initial opening gave a pressure drop with flow comparable to a 1-inch orifice, and the restriction then dropped to zero during the remaining opening time.

Figure 5.2 shows the calculated liquid oxygen flow rate during the initial portion of the cooldown period. A constant low flow occurs during the first 14 seconds, then increases rapidly to a maximum value at 41 seconds, and finally decreases to zero at 66 seconds. At 66 seconds, the check valve closes, preventing any backflow into the liquid oxygen storage vessel. Comparison of the flow curve with the maximum pressure curve shown on figure 5.1 illustrates the reason pressure surges develop during cooldown. The peak pressure occurs later than the maximum flow rate. The delay in the development of the downstream pressure allows additional liquid to be forced into the piping, thus increasing the magnitude of the pressure surge.



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Figure 5.2 Liquid Oxygen Flow, 14-Inch Line

5.2 Pressures Between the Inlet Valve (A1) and the Pump

The calculated pressure for the section of line between the inlet valve A1 and the pump is shown in figure 5.3. Again, the measured pressures are shown as a band and include data from several cool-downs. The measured pressures were obtained from sensor 2G07 located on the pump inlet line. The computer program did not predict the high pressure surge measured 4.5 seconds after the inlet valve was opened.

The inability of the computer to predict the high pressure surge raised doubts about the validity of the mathematical model. As time progressed, however, it became clear that the mathematical model was not capable of predicting the surge since it was caused, not by a cooldown phenomenon, but by a rapid deceleration of the inflowing liquid.

As stated during the description of the pressure surge phenomenon, pressure surges are generated when the rapidly flowing vapor entrains liquid downstream, increasing the heat transfer area causing larger amounts of vapor that must flow through the restriction of the vent system. The expansion of vapor beyond the check valve in the Kennedy system will not increase the pressure upstream of the check valve because the valve will close during any reverse flow. Even though a high restriction exists in the pump, this restriction is sufficiently small to allow all gas generated to be relieved at a reduced pressure.

Although many assumptions are necessary, pressure surges due to a quick deceleration of the liquid can be made. Salisbury [1950] gives an equation for the magnitude of the excess pressure due to water hammer in feet of head (h) of the fluid involved, the amount of sudden velocity reduction (U), and several other variables of the fluid and system. The equation is $h = AU/g_c$; where $A = U_s / \sqrt{1 + KB}$, U_s is sonic

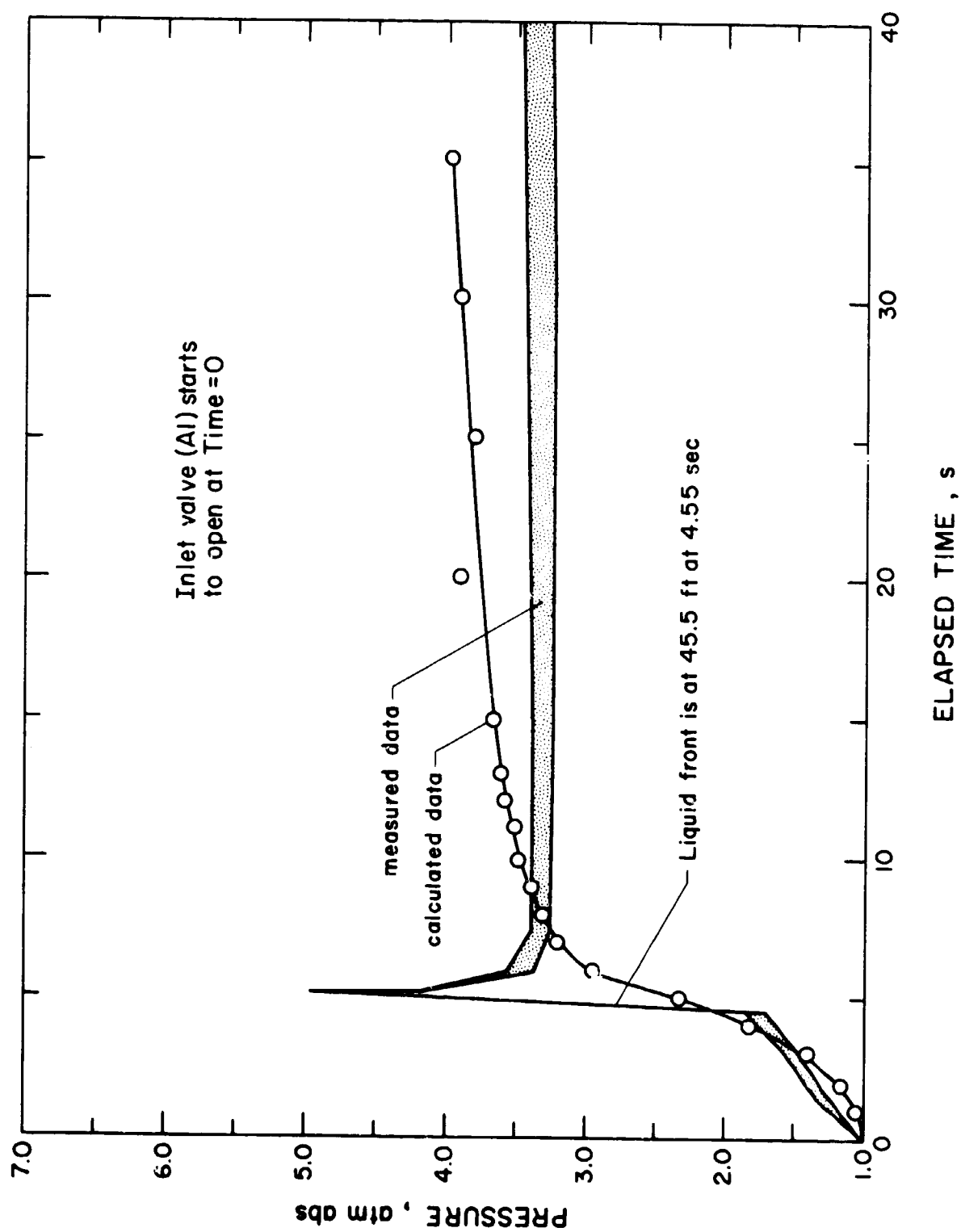


Figure 5.3 Pressures for Piping Between Inlet Valve (AI) and 10,000 GPM Pump.

velocity for the fluid involved, K is the ratio of the elastic moduli of the fluid to the material of the pipe shell, and B is the ratio of pipe diameter to thickness. The product KB is approximately 0.05 and will be assumed negligible, thus $A = U_s$.

The calculation of sonic velocity for the two-phase fluid present during the initial inflow was made using a correlation presented by Smith [1963]. Table 5.2 shows the calculated sonic velocities versus the qualities for two-phase oxygen.

Table 5.2
Sonic Velocity in Two-Phase Oxygen

Quality mass of vapor/total mass	Sonic Velocity ft/s
0	2637.8
.001	198.7
.03	107.6
.05	112.8
0.1	152.4
0.2	208.0
0.5	402.8
0.9	550.2
1.0	584.0

During the calculation for the section of pipe downstream of the check valve, the freewheeling pump had a restriction to flow comparable to a 1-inch diameter orifice during the initial stages of cooldown. Therefore, a 1-inch orifice plus two additional cooldown vents on the pump of 0.5-inch diameter were used to calculate initial liquid velocities. Just before the liquid-gas interface reaches the pump, the velocity of the interface (U_1) can be determined by,

$$U_i = U_{gi} - \frac{W}{\rho_{gi} A_f}; \quad (5.1)$$

where

U_{gi} = gas velocity at the interface,

W = mass rate of evaporation of the liquid,

ρ_{gi} = density of the gas at the interface,

A_f = flow area at the interface.

Assuming a step function in temperature at the interface from the liquid temperature to ambient temperature, $U_{gi} = U_s \left(\frac{A_o}{A_p} \right)$. U_s is sonic velocity in ambient temperature gas; A_o is the area of the orifice; A_p is the flow area of the pipe. $A_o = [(\pi/4)(1)^2 + 2(\pi/4)(0.5)^2] = 1.178 \text{ in.}^2$; $A_p = (\pi/4)(16.876)^2 = 223.68 \text{ in.}^2$; and sonic velocity is 1082.7 ft/s. Then $U_{gi} = 1082.7 (1.178/223.68) = 5.7 \text{ ft/s}$.

The mass rate of evaporation for the liquid (W) is determined by $W = A_s h / \Delta h_\ell$ where, A_s is the heat transfer surface area, h is the rate of heat transfer, and Δh_ℓ is the total enthalpy change from liquid to ambient temperature gas. Surface area, $A_s = 40.7 \text{ ft} \times \pi (16.876/12) \text{ ft} = 179.8 \text{ ft}^2$. The heat transfer rate corresponding to 2 W/cm^2 is $6341.2 \text{ Btu/h} \cdot \text{ft}^2$. The total change in enthalpy is 165.54 Btu/lb. Then, $W = 179.8 \times 6341.2 / 165.54 \times 3600.0 = 1.913 \text{ lbs/s}$. The density (ρ_{gi}) of the gas at the interface is $.3246 \text{ lb/ft}^3$, and $A_f = 1.553 \text{ ft}^2$. We can now determine the interface velocity from eq (5.1) as:

$$U_i = 5.7 - 1.913 / (.3246 \times 1.553) = 1.91 \text{ ft/s}.$$

Now, the velocity of the liquid after the interface enters the pump is calculated using Bernoulli's equation. The liquid velocity in the orifice is $U_o^2 = 2 g_c [U_\ell^2 / 2g_c + (P_1 - P_2) / \rho]$ for an incompressible fluid. Because the pipe is much larger than the orifice, U_ℓ is assumed zero;

U_0 becomes 38.92 ft/s. Calculating U in the pipe by using the area ratio again results in $U = 0.2$ ft/s. Therefore, the initial velocity in the liquid of 1.91 ft/s is almost completely quenched when the liquid front reaches the pump. Calculating the amount of pressure developed by this deceleration, assuming a zero quality results in $h = 2637.8 \text{ ft/s} \times 1.71 \text{ ft/s} / 32.2 \text{ ft/s}^2 = 140.1$ ft. of oxygen head or 65.6 psi. As noted from table 5.2, the sonic velocity at zero quality is the highest, so any two-phase regions in the liquid will cause a reduction in the calculated pressure. The measured pressure spike was off scale so a comparison between the calculated and measured data is not possible. The assumptions involved in the calculated pressure surge require that the results be interpreted only as an indication of a possible surge occurring and not the magnitude of the pressure surge.

Because the mathematical model did not calculate a pressure surge and because an explanation exists for the measured surge, it is assumed that the measured surge is caused by a deceleration phenomenon instead of a cooldown phenomenon.

6.0 COOLDOWN TIME

Two important factors controlling the cooldown time of pipes are the resistance to flow of vaporized liquid and/or the resistance to the transfer of heat from the wall to the cryogenic fluid. The cooldown behavior of any pipeline would lie between these extreme cases. If flow resistance is unimportant, the entire line could be filled with liquid in a relatively short time, and the temperature history at all stations along the line would coincide. This could be termed "heat transfer-controlled" cooldown and might be approached in small length-to-diameter ratio pipelines or in submerged bodies. The opposite extreme could be termed "flow controlled" cooldown where gas flow resistance is the important factor and resistance to heat flow is effectively zero.

Since cooldown time is important, a preliminary calculation of cooldown time for the Kennedy system has been made. Because of the high length-to-diameter ratio of the Kennedy system, a "flow controlled" cooldown was assumed valid for the system. Steward [1968] describes a simplified cooldown model using a "cold front" analysis. The model, however, assumes an insulated system and heat leak from the ambient is zero during the cooldown period. This model was modified to allow heat leak during the cooldown period and applied to the uninsulated Kennedy system.

6.1 Development of the Cooldown Model

Assumptions made during the development of the model were:

1. Temperature drop in the pipe is a step from ambient temperature down to the liquid inlet temperature. This temperature step, or cold front, advances down the pipe at a velocity, U_i .
2. Axial heat conduction is negligible.
3. A liquid-warm gas interface coincides with the pipe cold front at all times so that the interface velocity is also U_i .
4. Heat transfer in the gas stream is zero; hence, the upstream gas temperature is the initial wall temperature.
5. The pipeline is horizontal with no restrictions to flow or concentrated masses to cooldown.
6. The velocity changes are gradual enough that the process may be considered quasi-steady. Thus, the flow and pressure surges known to exist are smoothed out.
7. Because of the relatively low velocity of the liquid stream as compared to the low density, high velocity, warm gas stream, all the pressure drop is assumed to occur across the gas stream. Thus, the interface pressure is the same as the inlet pressure. Since the gas is warm, perfect gas

X_i = location of the upstream end of the gas stream, or liquid-vapor interface

$X_i = 0$ at the inlet end,

$K = hC$, where h is the heat transfer rate per unit area from ambient, and C is the circumferential length around the outside of the pipe.

The heat transfer can also be related to the amount of fluid evaporated by,

$$q = W(\Delta h_\ell), \quad (6.4)$$

where Δh_ℓ is the total cryogen enthalpy change from inlet conditions to ambient discharge conditions. Eliminating q , U_ℓ , and W from equations 6.1 through 6.4 gives an expression for the velocity of the liquid-vapor interface as,

$$U_i = \frac{U_{gi}}{B_1} - \frac{X_i B_2 U_s}{L B_1}, \quad (6.5)$$

where $B_1 = 1 + \frac{\rho_w A_w \Delta h_w}{\rho_{gi} A_f \Delta h_\ell}$, and $B_2 = \frac{KL}{\rho_{gi} A_f \Delta h_\ell U_s}$.

According to the assumptions, B_1 and B_2 are constant and depend only on the beginning and end conditions.

The cooldown time, T , is defined as the time needed for the interface to travel the length of the pipe, or

$$T = \int_0^L \frac{1}{U_i} dX_i. \quad (6.6)$$

relationships are used for the vapor phase. The ratio of specific heats was taken as 1.4 in the calculations.

The velocity of the liquid-gas interface is the liquid inflow velocity minus the velocity at which the liquid front evaporates, or

$$U_i = U_l - \frac{W}{\rho_l A_f}, \quad (6.1)$$

where

U_i = interface velocity,

U_l = liquid inflow velocity,

W = mass rate of evaporation,

ρ_l = density of the inflowing liquid,

A_f = flow cross section area.

The upstream gas velocity, U_{gi} , can be found from

$$U_{gi} = U_i + \frac{W}{\rho_{gi} A_f}, \quad (6.2)$$

where ρ_{gi} is the density of the gas at the liquid-vapor interface.

The total heat transferred to the liquid cryogen (q) is the sum of the heat transferred from ambient and the heat removed from the pipe wall and may be expressed as;

$$q = U_i \rho_w A_w \Delta h_w + KX_i, \quad (6.3)$$

where

ρ_w = density of the wall material,

A_w = solid area of the pipe walls,

Δh_w = total change in enthalpy of the wall from initial conditions to the final cooldown condition,

Substituting eq (6.5) into eq (6.6), a dimensionless group of numbers can be obtained:

$$\frac{TU_s}{B_1 L} = \int_0^1 \frac{d\left(\frac{X_i}{L}\right)}{M_i - \frac{X_i B_2}{L}}, \quad (6.7)$$

where, U_s is sonic velocity in the ambient temperature gas phase, and M_i is the Mach number of the upstream gas. Using the Fanno relationship for the gas stream pressure drop and selecting a pressure ratio, a friction factor, and a B_2 , eq (7) was numerically integrated using a computer. A more complete description of the integration procedure and the use of the Fanno relationship is given by Steward [1968].

Figures 6.1, 6.2, 6.3, and 6.4 show the results of the computer solution. Each figure is for a selected pressure ratio and gives a time parameter for various B_2 's and fL/D 's. When B_2 is zero, the heat leak from ambient is zero.

6.2 Cooldown Time for the KSC System

Again, because of the simplifications made in the cooldown model, an equivalent system must be defined to correctly determine cooldown time for the Kennedy system. The vertical rise in the oxygen system will effectively decrease the pressure ratio available to affect the final stages of cooldown. The total vertical rise in the oxygen system is approximately 230 feet creating a total pressure head of 107 psi when the vertical section is completely filled with liquid oxygen. Calculations of cooldown time will be made assuming an upstream pressure of three times the discharge pressure ($P_r = 3$). Actual pressures required at the inlet of the line will increase from an initial 3 atmospheres to a final 8.3 atmospheres during the cooldown period.

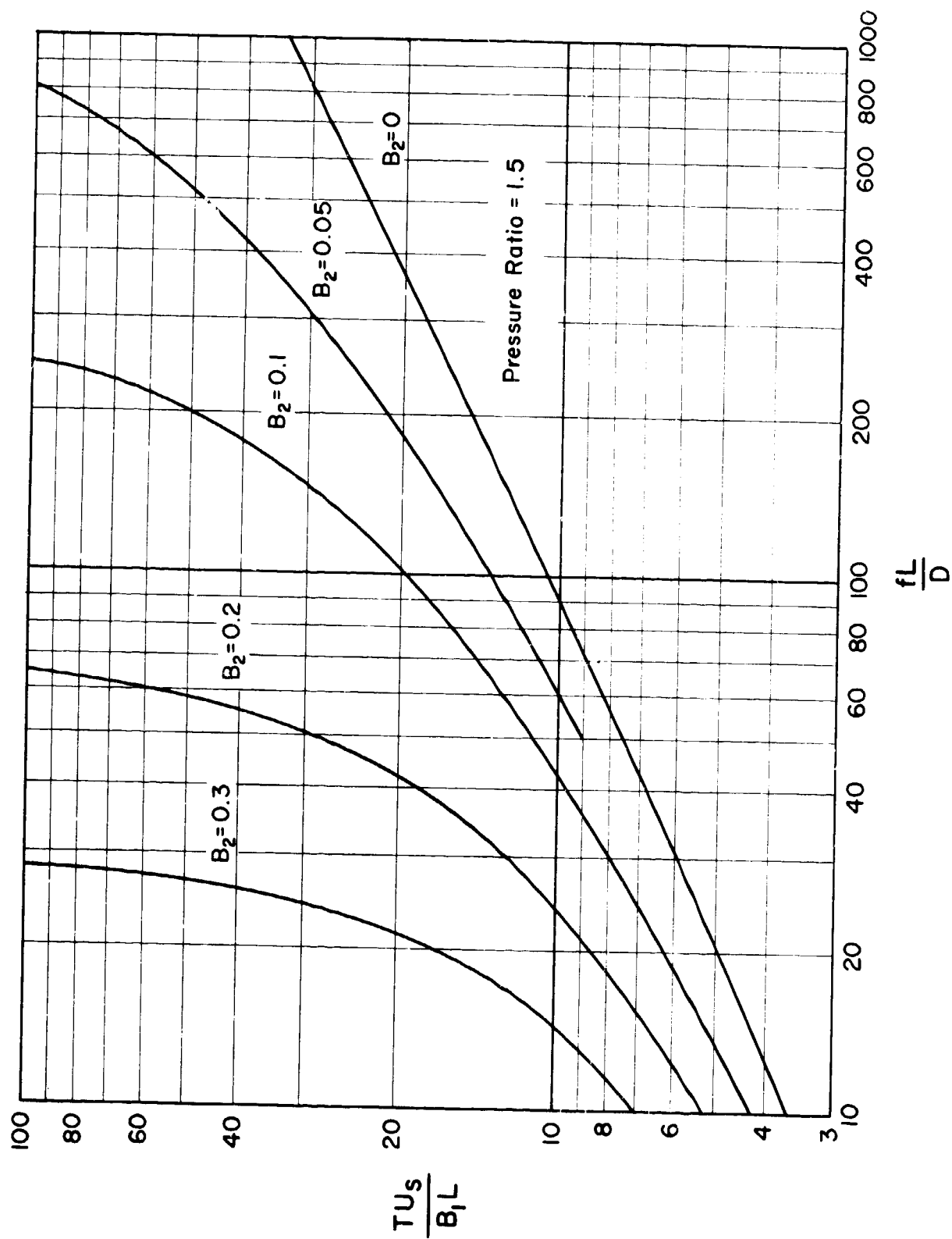


Figure 6.1 Cooldown Time Parameter for Lines with Heat Leak ($Pr = 1.5$).

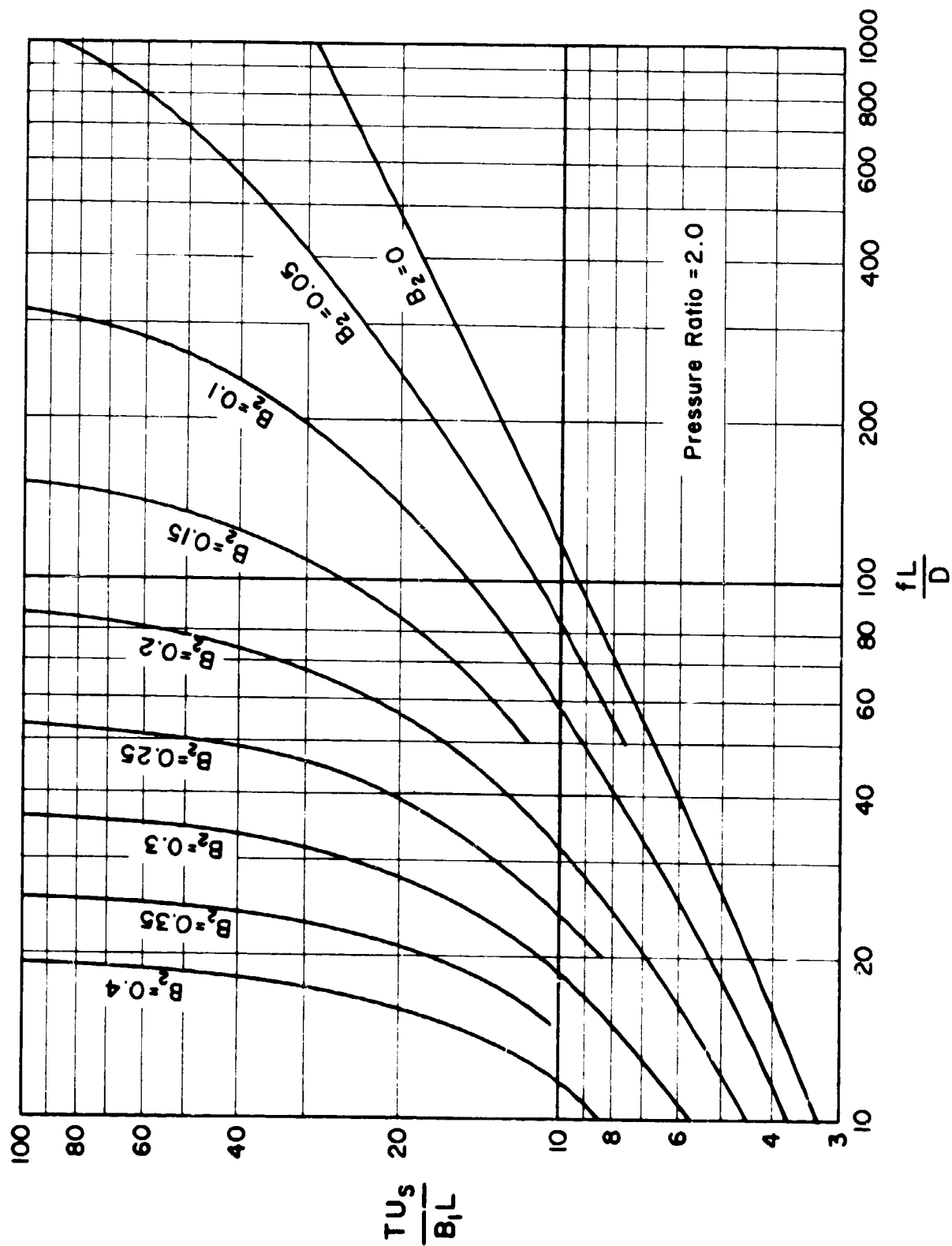


Figure 6.2 Cooldown Time Parameter for Lines with Heat Leak ($Pr = 2.0$).

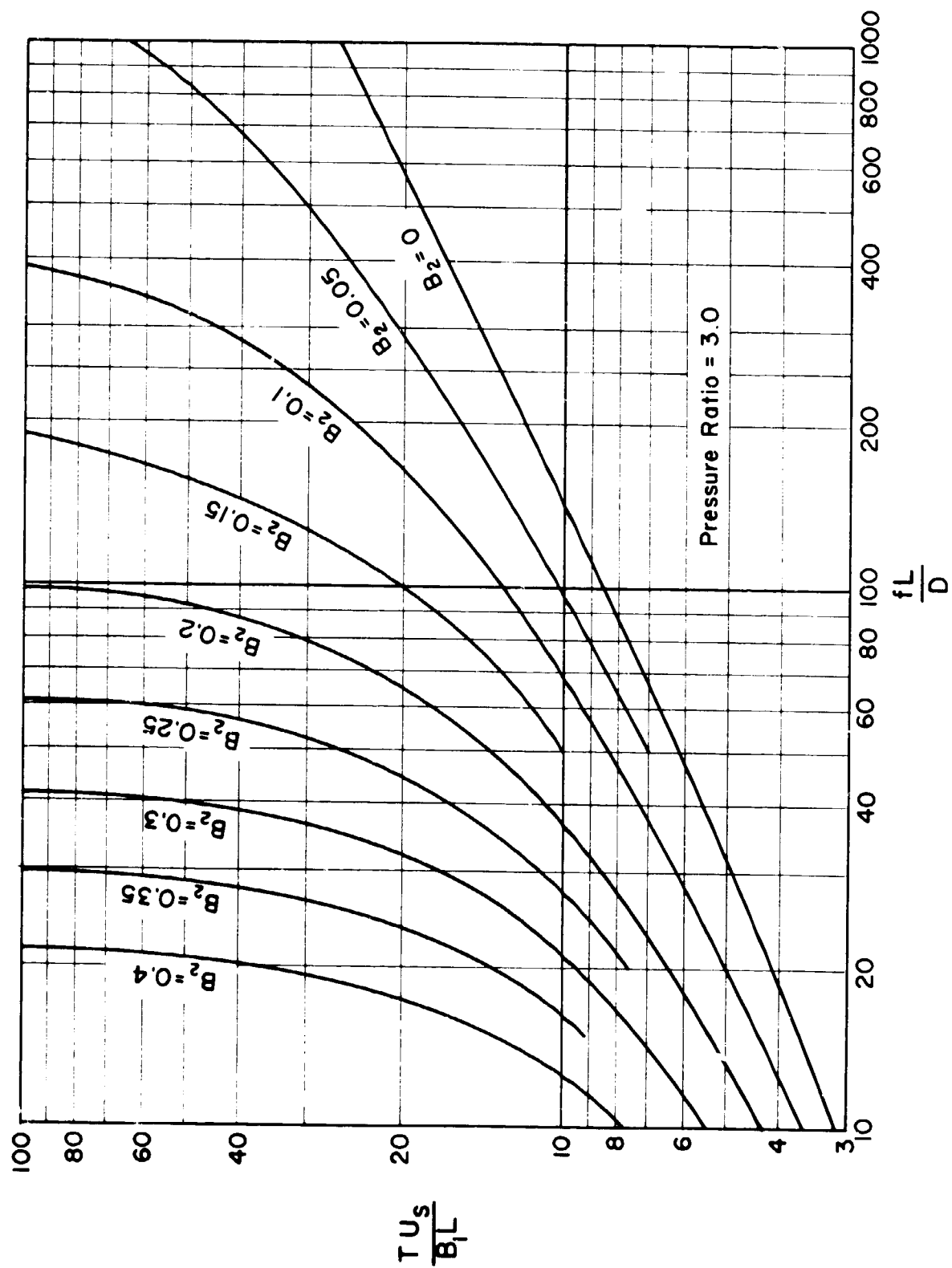


Figure 6.3 Cooldown Time Parameter for Lines with Heat Leak ($Pr = 3.0$).

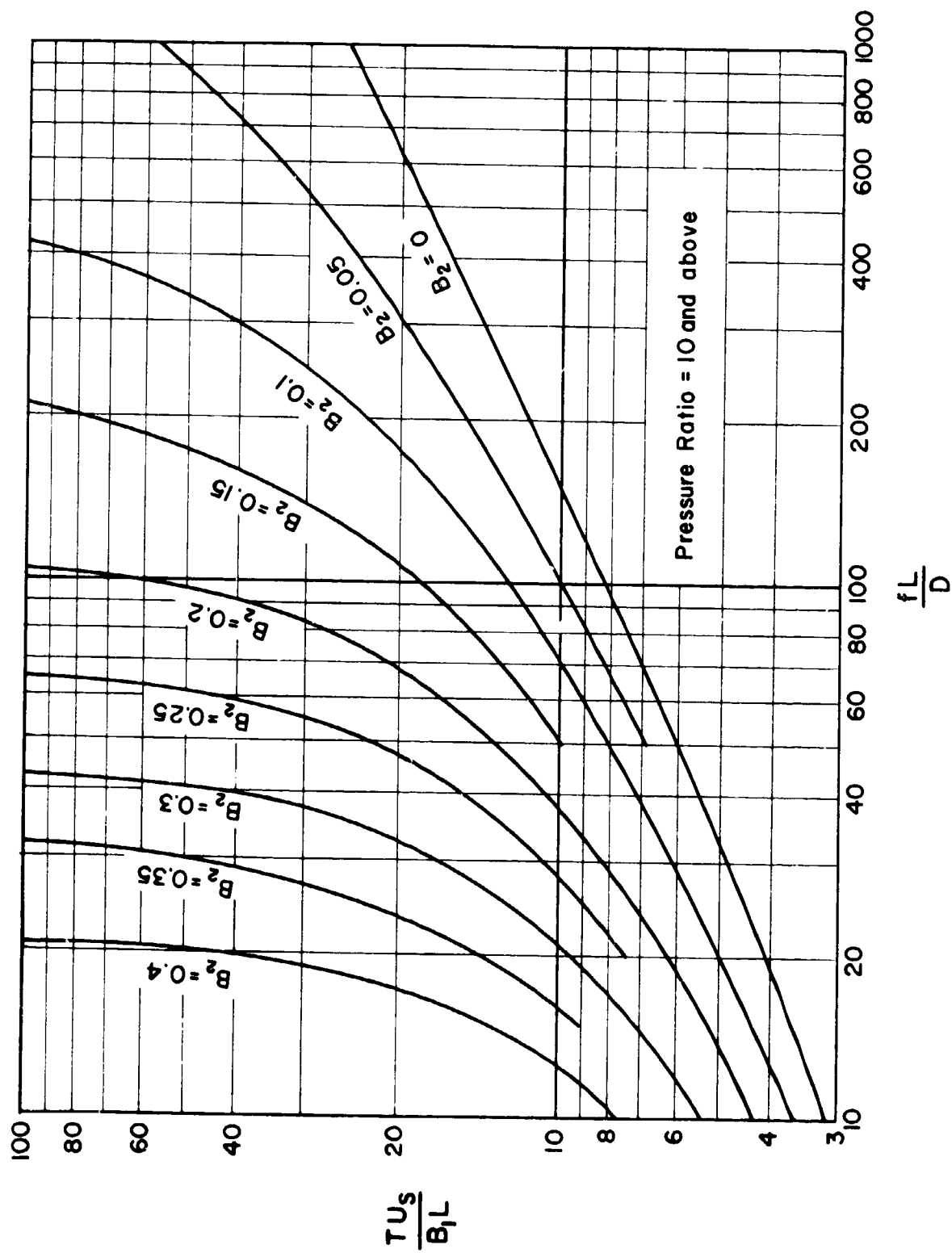


Figure 6.4 Cooldown Time Parameter for Lines with Heat Leak ($Pr = 10, 0$ and above).

The major complication in the system is the decrease in line size occurring on the launch umbilical tower. Because of this decrease, an equivalent constant diameter line length was determined for the system. Equivalent system length was determined by equating the gas pressure drop for the actual system with the pressure drop in an equivalent system. The best equivalent system had a diameter equal to the smallest diameter in the system with the larger diameter line length decreased by the ratio of $(D_S/D_L)^5$. The thickness (and resultant mass) of material in the wall and the heat transfer to the wall from the ambient was increased by the inverse of the diameter ratio to the fifth power to compensate for the decreased length. Other restrictions such as valves, elbows, and tees were simulated by adding equivalent lengths of pipe for the component.

Another important variable was the heat transfer rate between the ambient and the line during cooldown. The heat transfer rate between a cryo-surface and ambient air is discussed by Ruccia [1958], Holten [1960], Richards [1962], and Smith [1963]. The heat transfer rate appears to depend on the time the cryosurface has been cold, the moisture content of the ambient air, and wind velocity. The rates shown in the references vary from a high of 7000 Btu/h-ft^2 at high wind velocities of 55 miles per hour and high relative humidities to a low of 480 Btu/h-ft^2 for a vertical pipe protected from wind in air of an unknown relative humidity. It would be expected that the heat transfer rate would be high during the initial stages of cooldown because the insulating layer of frost would not be present and decrease to a steady state value in approximately 2 hours. Since the exact heat transfer rate can only be estimated, cooldown time for various heat transfer rates was calculated to show their effect.

An average friction factor (f) for the gaseous portion of the cooldown flow must also be defined. The method described by Steward [1968] was used to define an average friction factor. The friction factor used was based on the initial gas velocity U_{gi} and was held constant during the calculation.

Cooldown time was calculated for the complete length of line, including the vent system, and then the cooldown time for the 10-inch vent section was subtracted leaving the final cooldown time for only the line up to the stage inlet valve.

Figure 6, 5 shows the results of calculations for various heat transfer rates. Results are presented for two pressure ratios applied to the equivalent system and a single pressure ratio applied to an altered system. The altered system was a pipeline of 14-inch constant diameter venting at the inlet valve to the stage. The real unknown becomes the heat transfer rate from ambient to the uninsulated line. The difficulty in determining the heat transfer rate is due in part to the transient nature of the cooldown phenomenon and to the dependence of the heat exchange rate on wind and air moisture content. However, the general effects of the heat transfer rate can be noted from the curves. Also, if a higher pressure ratio or larger vent area is available, the cooldown time becomes less dependent upon the heat leak. Because of the assumptions involved, actual cooldown times are difficult to determine accurately; however, if the 10,000 gpm pump can be started early in the cooldown period, an average pressure ratio of more than three can be maintained on the system, reducing cooldown times to less than those shown by the upper curve. Since the system involves a vertical section, maintaining a pressure ratio of 10 during the entire cooldown period would be difficult; so cooldown times would be longer than those shown by the lower equivalent model curve. Thus, actual cooldown time

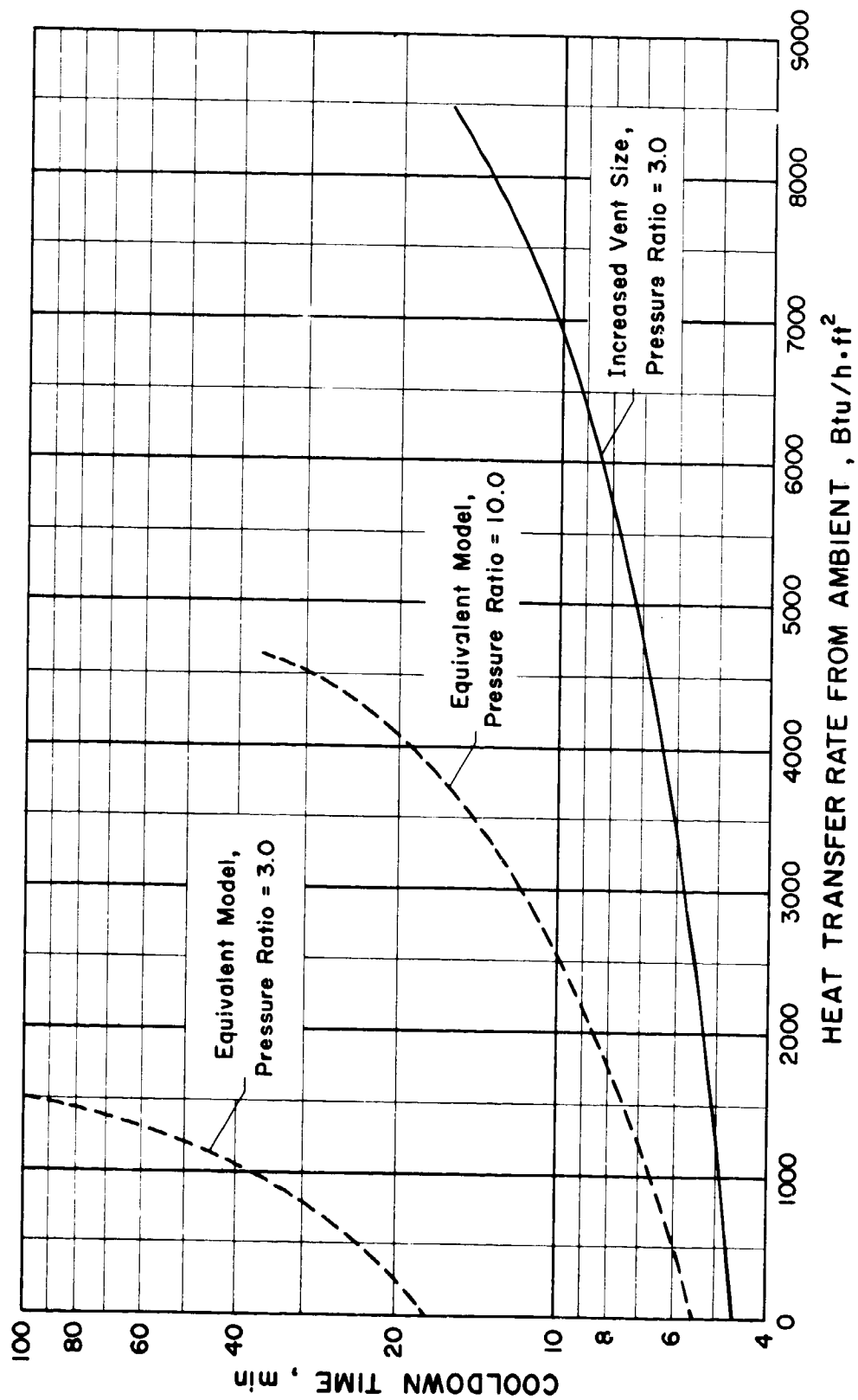


Figure 6.5 Cooldown Time for 14-Inch Cross Country LOX Line.

would be between the two curves, probably closer to the lower equivalent model curve. Major decreases in cooldown times could be achieved by shortening and increasing the size of the vent system as shown by the lowest curve.

7.0 SUMMARY AND RECOMMENDATIONS

Pressure surges developed during cooldown, although not an insignificant problem, can be eliminated or minimized, thus reducing the damage potential of the surges. An experimental apparatus by Steward of NBS was specifically designed to create pressure surges and to determine the factors influencing the magnitude of the surges. The primary factor influencing the magnitude of pressure surges, as determined by Steward [1968], was the amount of subcooling present in the initial inflow of liquid. During his work, the liquid nitrogen in the storage vessel, located at the inlet of the line, was saturated at one atmosphere, and pressure surge data were taken by increasing the storage vessel pressure just prior to opening the inlet valve. Higher storage vessel pressures resulted in higher pressure surges. With the liquid nitrogen in the storage vessel at 5 atm, measured peak pressures were approximately 36 atm or a factor of 7 times the inlet pressure. When the driving pressure in the storage vessel was 3.4 atm, the measured peak pressure surge was reduced to 6.9 atm or only a factor of two above the driving pressure. Steward also found that the measured peak pressures decreased even further if the liquid was allowed to warm even slightly above the one atm saturation temperature prior to initiation of cooldown.

A second factor having an influence on the magnitude of cooldown pressure surges was the inlet valve opening time. Maximum pressure surges with nitrogen were measured when the inlet valve opening time was approximately 0.5 second or less. In Steward's experiment, the time necessary for the first surge to develop was 4 seconds. This time compares favorably with the calculated time for the initial pressure

surge to occur in that portion of the Kennedy system between the inlet valve A1 and the pump; however, the inlet valve on the Kennedy system opens in 5 seconds (rather than 0.5 seconds), further indicating that pressure surges should be reduced in this portion of the system.

Steward discovered several other factors that had minor influences on the magnitude of pressure surges. The mathematical model used in this study included these factors, but very little change in the calculated peak pressure could be attributed to these factors.

The check valve located downstream of the pump could produce large pressure surges downstream of the check valve except for the relatively warm temperature of the liquid going through the pump and the apparent small, initial, effective flow area of the freewheeling pump. Even so, measured and calculated pressure surges downstream of the check valve were on the order of 3 times the supply pressure.

Preliminary cooldown time calculations indicate a relatively long cooldown period for the present system, particularly at high heat transfer rates from the ambient. Decreases in cross-country line cooldown time can be achieved if the pump can be started early in the cooldown period, or if the smaller 10-inch line on the discharge end of the cross-country line is replaced with a 14-inch line; starting the pump early and modifying the plumbing results in still shorter cooldown times.

One possible method for starting the 10,000 gpm pump early in the cooldown period is to precool the pump prior to cooldown of the cross-country line. The procedure would be as follows:

1. The section of line between the tank and the inlet valve A1 would be cooled in the present manner; that is, valve A109 would be opened for an extended period of time before valve A1 is opened.

2. The pump loop between valve A1 and the storage tank would be cooled. This loop includes the dashed line on figure 3.1. Valves A28756 and A102 would be closed and valves A113, A112, and finally valve A1 would be opened. Valves A28707 and A28706 could also be opened a short time if necessary. The liquid oxygen head in the storage tank would force liquid oxygen through the pump with the vapor returning to the tank ullage. The ullage pressure in the storage tank would remain at one atmosphere, being relieved through the normal tank vent. The storage tank ullage would act as a ballast volume absorbing any cooldown pressure surges generated during this period. Sufficient time would be allowed in this configuration to completely cool and fill the pump with liquid oxygen.

3. Valve A28756 would be opened allowing the cooldown of the cross country line. Valve A102 would be opened, valve A103 closed, the storage tank ullage pressurized, and then the 10,000 gpm pump could be started. Again, the storage vessel would act as a ballast volume relieving any cooldown pressure surges occurring during this period.

4. Valve A102 would be slowly closed diverting the entire pump output into the cross-country line. Cooldown would now proceed rapidly because of the increased driving pressure from the pump.

5. Completion of cooldown could be determined by observing the pressure head developing in the vertical stand pipe on the launch umbilical tower. When the liquid head indicates the stand pipe is filled with liquid, the flow could be diverted into the oxidizer tank.

Pre-chilling the pump loop (steps 1 and 2) would decrease the length of time when significant oxygen venting would occur near the launch area. Venting near the launch area would not occur until the launch area had been cleared and step 3 was initiated. Then cooldown of the cross-country line, following the procedure outlined in steps 3, 4, and 5 could be completed in 10 to 15 minutes according to the $Pr = 10$ curve on figure 6.5. Also, sufficient time could be allowed in steps 1 and 2 to insure a complete cooldown of the pump, removing any temperature gradients which may damage the pump due to a premature startup.

The proposed cooldown procedure requires further study before it can be recommended; however, if quick cooldown of the cross country line is required, this plan could be implemented without appreciable changes to the existing plumbing or control systems. In addition, modification of the plumbing (10 to 14-inch) would further decrease cooldown time.

Recommendations for continuing work in the calculation of cooldown pressure surges would include the following:

1. Extend the mathematical model to include the generation of pressure surges due to a sudden deceleration of the liquid oxygen within the pipeline.
2. Construct and program a computer package to simulate the KSC liquid oxygen loading system. Make the computer package sufficiently flexible allowing for system and cooldown procedural changes.

8.0 CONCLUSIONS

The following conclusions were made based on the study:

1. The initial cooldown pressure surge in a 14-inch line can be predicted using a simplified mathematical model. Therefore, changes in the system and cooldown procedure can be investigated mathematically, saving the expense of experimental testing.
2. A water hammer type of pressure surge occurs in the line between the inlet valve A1 and the pump during the current cooldown procedure. Otherwise, no high or damaging surges occur in the piping system.
3. Cross-country line cooldown time can be reduced significantly if the 10,000 gpm pump can be started early in the cooldown period; cooldown time can be accelerated even more by increasing the diameter of the discharge pipe to 14-inches. A possible method for starting the pump early is to precool the pump before cooldown of the cross-country line is initiated. This method also allows the use of the storage tank ullage as a surge chamber, reducing the possibility of damage due to pressure surges during cooldown.

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APPENDIX I. Cooldown computer program listing

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PROGRAM COOLDOWN
C  COOLDOWN SURGE A1
10 DIMENSION XL(200),PXL(200),VL(200),XQ(200),DT(200),PG31(200)
11 DIMENSION TIM(200),XVAP(200),TL(200),XPREV(200),TPREV(200)
12 DIMENSION XR(200),NB(200),NPREV(200)
54 FORMAT(6F10.0,E12.4)
312 FORMAT(4E12.4,2F10.0)
313 FORMAT(8F10.4)
314 FORMAT(3F10.0,3E12.4,2I5)
53 READ 54,DELTX,TON,XCOLD,X4,DIAM,QFACT,PIN
   IF(DELTX.EQ.0.0) GO TO 312
   READ 312,PXLO,PAMB,C5,C6,XVALV,DENSL
   READ INPUT TAPE 5,313,A,B,C1,C2,C3,C8,C9,C10
   READ 314,C11,FR,TIN,V SOUND,C12,C13,IPRINT,ISTOP
541 WRITE OUTPUT TAPE 6,542
542 FORMAT(1H1,42H COOLDOWN SURGE A1 ,OXYGEN , INPUT DATA  //)
543 WRITE OUTPUT TAPE 6,544
544 FORMAT(8H      PIN,8X,4HDIAM,5X,2HX4,5X,5HXCOLD,4X,5HXVALV,3X,
13HTON,3X,5HDELTX,4X,5HDENSL,4X,6HVSOUND,7X,3HTIN,7X,4HPAMB/
212H DYNE/SQ CM,5X,2HCM,
36X,2HCM,6X,2HCM,7X,2HCM,5X,3HSEC,4X,2HCM,4X,8HGM/CU CM,3X,
46HCM/SEC,6X,5HDEG K,3X,10HDYNE/SQ CM)
545 WRITE OUTPUT TAPE 6,546,PIN,DIAM,X4,XCOLD,XVALV,TON,DELTX,DENSL,
1VSOUND,TIN,PAMB
546 FORMAT(E12.4,F8.3,F9.1,F8.1,F8.1,F8.3,F7.1,F9.4,E13.4,F8.2,
1E13.4//)
547 WRITE OUTPUT TAPE 6,548
548 FORMAT(5H      A,7X,1HB,7X,2HC1,6X,2HC2,7X,2HC3,9X,2HC5,11X,2HC6,
111X,2HC7,11X,2HC8,9X,2HC9,7X,3HC10,6X,3HC11)
      C7=1.0
549 WRITE OUTPUT TAPE 6,551,A,B,C1,C2,C3,C5,C6,C7,C8,C9,C10,C11
551 FORMAT(F7.3,F8.3,F8.1,F9.4,F9.4,4E13.4,F9.4,F9.4,F9.2//)
5520 WRITE OUTPUT TAPE 6,5521
5521 FORMAT(5X,3HC12,10X,3HC13,9X,2HFR,3X,5HQFACT,5X,5HDIAMG,5X,5HDIAML
1,5X,4HX4EQ,5X,5HXPUMP)
5522 PRINT 5523,C12,C13,FR,QFACT,DIAMG,DIAML,X4EQ,XPUMP
5523 FORMAT(E12.4,E13.4,F8.3,F7.2,3X,F7.2,3X,F7.2,2X,F7.1,3X,F7.1//)
554 WRITE OUTPUT TAPE 6,555
555 FORMAT(9H RESULTS//9X,5HXL(I),4X,6HTIM(I),6X,6HPXL(I),8X,5HPVALV,
19X,3HPFL,10X,3HPG3,9X,5HVL(I),4X,3HGM3,6X,3HGM4,7X,5HSUMXQ,3X,
24HICTR/6H      CM,7X,3HSEC,6X,10HDYNE/SQ CM,3X,10HDYNE/SQ CM,4X,
310HDYNE/SQ CM,3X,10HDYNE/SQ CM,4X,6HCM/SEC,24X,2HCM//)
64 I=1
   J=2
65 XL(1)=XVALV
66 PXL(1)=PXLO
67 VL(1)=0.000001
68 PVALV =PIN-PAMB
69 PG3=PAMB
691 GM3=0.0
   TSAT=TIN
692 GM4=0.0
   XCOLD=XCOLD+XVALV
693 ICTR=0.0

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```

70 TIM(1)=0.0
   TL(1)=TIM
   XQ(1)=0.0
   SUMXQ=0.0
   XR(1)=0.0
   C14=1.0
71 PL3=PAMB
73 SIGN=-1.0
76 WRITE OUTPUT TAPE6,761,XL(I),TIM(I),PXL(I),PVALV,PFL,PG3,VL(I),
   IGM3,GM4,SUMXQ,ICTR
761 FORMAT(1X,F13.2,F9.4,E14.4,E13.4,E14.4,E13.4,F10.2,F9.4,F9.4,F11.2
   1,I5)
   IF(SIGN)7611,7611,7600
7611 IF(XL(I)-XCOLD)7612,7612,7600
7612 IF(I-25)7600,7600,311
7600 DO 7603 K=1,I
   XPREV(K)=XR(K)
   NPREV(K)=NB(K)
   IF (TL(K)-TSAT)7603,7601,7601
7601 XSAT=XL(K)
   IF(K-5)7603,7603,7602
7602 C14=QFACT
7603 TPREV(K)=TL(K)
   IF(I.LT.15)GO TO 762
   DELTX=ABSF(XL(I)-XL(J))+10.0
   IF(DELTX.GT.200.0) DELTX=200.0
762 I=I+1
   NOTE=-1
   NOTE2=-1
78 ICTR =0
79 J=I-1
   IF(I.EQ.2) GO TO 85
82 IF (VL(J))91,85,91
85 IF(NOTE)86,86,91
86 SIGN=-1.0*SIGN
91 XL(I)=XL(J)+(SIGN*DELTX)
   IF(SIGN.LT.0.0.AND.XL(I).LT.XVALV ) GO TO 311
77 TALLY=0.0
781 JCTR =0
   NBEST=-1
782 KCTR = 0
783 LCTR=0
94 PXL(I)=PXL(J)
95 GO TO 99
97 ICTR = ICTR +1
   IF(KCTR-25)98,98,971
971 PXL(I)=PXSAVE
   ERROR=(PG3R-PG3)/PG3
   PRINT 972,ERROR
972 FORMAT(2X,*ERROR=*,E20.5,2X,*EXCEEDS TOLERANCE*)
   NBEST=1
98 IF (ICTR - 40)99,99,1000
99 RAD=((XL(J)/XL(I))*(VL(J)*VL(J))+(((1.0-XL(J)/XL(I))*2.( DENSL)))*
   1(PXL(I)+PXL(J))/2.0)

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```

100 IF(RAD)135,135,101
101 VL(I)=SIGN*SQRTF(RAD)
106 DT(I)=2.0*(XL(I)-XL(J))/(VL(I)+VL(J))
109 TIM(I)=TIM(J)+DT(I)
    IF(TIM(I).GT.TON) GO TO 121
    X=20.0*TIM(I)
    IF(TIM(I).LT.0.2) X=4.0
    Z=100.0+.212*X-.00212*X**2-(10000.0-X**2)**0.5
    Z=Z*655.58/100.0
    PVALV=1.067*(VL(I)/Z)**3.46/14.696*1.01325E6
118 GO TO 124
121 PVALV=0.0
124 REL=C1*SIGN*VL(I)
127 FFL=A/(REL**B)
    IF(SIGN)130,130,128
128 IF(XL(I)-XCOLD)130,130,1331
130 PFL=SIGN*C2*FFL*XL(I)*(VL(I)**2 )
133 GO TO 148
1331 IF(XL(J)-XCOLD)1332,1332,1333
1332 XSAT=XL(I)
1333 PFL=SIGN*C2*FFL*XSAT*VL(I)*VL(I)
    GO TO 148
135 IF(TALLY)137,137,1351
1351 XL(I)=(XL(I)+XL(J))/2.0
1352 GO TO 77
137 PXL(I)=-((DENSL*XL(J)*VL(J)*VL(J))/(XL(I)-XL(J))-PXL(J)
139 VL(I)=1.0
140 DT(I)=2.0*(XL(I)-XL(J))/VL(J)
141 TIM(I)=TIM(J)+DT(I)
142 PVALV=0.0
143 REL=0.0
144 FFL=0.0
145 PFL=0.0
146 TALLY=1.0
148 PL3=PIN-PFL-PVALV-PXL(I)
201 PG3=PL3
    IF(PG3)2011,2011,202
2011 IF(NOTE2-10)2014,2014,2012
2012 WRITE OUTPUT TAPE 6,2013
2013 FORMAT(18H NEGATIVE PRESSURE)
    GO TO 311
2014 PXL(I)=PIN-PAMB-PFL-PVALV
    NOTE2=NOTE2+1
    IF(I.GT.2) GO TO 2015
    IF(PXL(I).LT.0.0) PXL(I)=1.0
2015 GO TO 97
202 TSAT=76.89597+.18215E-4*PG3-.06417E-10*PG3*PG3+.15665E-17*PG3*PG3
    1*PG3-.20747E-24*PG3*PG3*PG3*PG3+.112364E-31*PG3*PG3*PG3*PG3*PG3
149 IF (SIGN)154,154,150
150 IF(XL(I)-XCOLD)152,152,155
152 XQ(I)=0.0
    XR(I)=0.0
    SUMXQ=0.0
    NB(I)=-1

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```

      TL(I)=TIN
      GO TO 190
154  XQ(I)=XL(I)-XL(J)
      NB(I)=-1
      XR(I)=XL(I)-XL(J)
      GO TO 165
155  IF(TIN-TSAT)1551,1551,1552
1551 XFL=0.0
      GO TO 156
1552 XFL=C12*(XL(I)-XL(J))*(TIN-TSAT)/PG3
156  IF(XPREV(J))157,157,159
157  M=I
159  TNB=TIN+C13*(VL(I)*PG3/(TIN      ))**0.8*DT(I)*C14
      XVAP(I)=(XL(I)-XL(J))
      IF(TNB-TSAT)163,163,161
161  TL(I)=TSAT
      NB(I)=1
162  XQ(I)=(XL(I)-XL(J))*(PAMB*(1.0-FR)+PG3*FR)
      1/(PAMB*(1.0-FR)+PG3*FR-PAMB*C3*DT(I))+XFL
      XR(I)=XQ(I)
      GO TO 165
163  XQ(I)=0.0
      XR(I)=XL(I)-XL(J)
      NB(I)=-1
      TL(I)=TNB
165  SUMXQ=XQ(I)
      IF(M-J)166,166,190
166  IF(I-IPRINT)1661,1662,1661
1661 IF(NOTE)1664,1664,1662
1662 WRITE OUTPUT TAPE 6,1663
1663 FORMAT(4H      K,6X,3HTNB,8X,1HY,8X,1HR,6X,5HXQ(K),3X,5HNB(K))
1664 DO 181 K=M,J
      IF (XPREV(K)) 167,167,169
167  XQ(K)=XPREV(K)
      XR(K)=XQ(K)
      GO TO 177
169  IF(NPREV(K))170,170,173
170  TNB=TPREV(K)+C13*ABSF(VL(I)*PG3/(TIN))*0.8*DT(I)*C14
      IF(TNB-TSAT)171,171,173
171  XQ(K)=0.0
      XR(K)=XPREV(K)
      TL(K)=TNB
      NB(K)=-1
      GO TO 177
173  Y=(XPREV(K)-XVAP(K))*(PG3*FR+PAMB*(1.0-FR))/(XVAP(K)*C11*PAMB)
      R=EXP(-12.4*(Y**1.715-0.254)**2)
174  XQ(K)=XPREV(K)*(PAMB*(1.0-FR)+PG3*FR+C3*DT(I)*PAMB*R)
      1/(PAMB*(1.0-FR)+PG3*FR-C3*DT(I)*PAMB*R)
      IF(XQ(K).LT.0.0) GO TO 1351
      TL(K)=TSAT
      XR(K)=XQ(K)
      NB(K)=1
177  IF(I-IPRINT)178,179,178
178  IF (NOTE)181,181,179

```

```

179 WRITE OUTPUT TAPE 6,180,K,TNB,Y,R,XQ(K),NB(K)
180 FORMAT(I5,F10.3,F9.4,F9.4,F10.2,I5)
181 SUMXQ=SUMXQ+XQ(K) *R
190 VG3= VL(I)+C5*SUMXQ/PG3
205 GM3=VG3/V SOUND
208 REG=C6*PG3*VG3
211 FFG=A/(REG**B)
214 FLOD=FFG*(X4-XL(I))/DIAM
220 FM4=1.1667*FLOD-(0.8333/(GM3**2 ))-LOGF(GM3**2 /(1.0+0.2*GM3
1**2 ))
2201 IF(I-IPRINT)2202,221,2202
2202 IF(NOTE )223,223,221
221 WRITE OUTPUT TAPE 6,222,XL(I),PXL(I),RAD,VL(I),TIM(I),PVALV,REL,
1FFL,PFL,PL3,XQ(I ),SUMXQ,PG3,GM3,REG,FFG,FLOD,FM4,TSAT,M,XFL,XSAT
222 FORMAT (8H XL(I)=,F7.2,3X,7HPXL(I)=,E12.5,3X,4HRAD=,E12.4,3X,4
1HVL=,F8.2/8H TIM(I)=,F6.4,3X,6HPVALV=,E12.4,3X,4HREL=,E12.4,3X,
24HFFL=,F7.4,3X/5H PFL=,E12.4,3X,4HPL3=,E12.4,3X,8HXQ(I )=,F7.2,3X
3,6HSUMXQ=,F7.2/5H PG3=,E12.4,3X,4HGM3=,F6.4,3X,4HREG=,E12.4,3X,
44HFFG=,F7.4,3X,5HFLOD=,F8.3/5H FM4=,F9.3,3X,5HTSAT=,F8.3,3X,2HM=,
5I3,3X,4HXFL=,F8.2,3X,5HXSAT=,F8.2)
223 IF(FM4+0.651)233,233,224
224 JCTR=JCTR+1
2241 IF(JCTR-2)2242,2244,2251
2242 FM4B = FM4
2243 PXB = PXL(I)
226 PXL(I)=PXL(I)-(FM4+0.651)*C8
229 GO TO 97
2244 PXFC=PXL(I)
FSAVE =FM4
PFSAVE=PXL(I)
2245 PXL(I)=PXL(I)-(FM4+0.6515)*(PXL(I)-PXB)/(FM4-FM4B)
2246 FM4C=FM4
2248 GO TO 97
2251 PXE=PXL(I)
IF(FSAVE-FM4)2254,2254,2252
2252 PFSAVE=PXL(I)
2253 FSAVE=FM4
2254 IF(KCTR-2)2256,2255,2255
2255 IF(ABSF((PXSAVE-PFSAVE)/PXSAVE)-.001)971,971,2300
2300 IF(KCTR-5)2256,2256,2301
2301 PXL(I)=(PXSAVE+PFSAVE)/2.0
GO TO 97
2256 Y1=(FM4C+0.6515)*(FM4B+0.6515)*(PXB-PXFC)
Y2=(FM4+0.6515)*(FM4B+0.6515)*(PXB-PXL(I))
Y3=(FM4+0.6515)*(FM4C+0.6515)*(PXFC-PXL(I))
PXL(I)=(PXL(I)*Y1-PXFC*Y2+PXB*Y3)/(Y1-Y2+Y3)
PXFC=PXE
FM4C=FM4
GO TO 97
233 GM4=1.0/(0.891*((-FM4-0.651)**0.5229)+1.0)
236 FM4T=LOGF(1.0/(GM4**2 )+0.2) -0.8333/(GM4**2 )
242 IF(ABSF((FM4T-FM4)/FM4)-0.001)254,254,245
245 DFM4=-2.0/(GM4+0.2*(GM4**3.0))+1.667/(GM4**3.0)
248 GM4=GM4+(FM4-FM4T)/DFM4

```

```

251 GO TO 236
254 P3OP4=GM4/GM3*SQRTF((1.0+0.2*(GM4**2 ))/(1.0+0.2*(GM3**2 )))
PG3R=PAMB*P3OP4
IF(I-IPRINT)255,256,255
255 IF (NOTE)257,257,256
256 WRITE OUTPUT TAPE 6,2561,GM4,P3OP4,PG3R
2561 FORMAT(6H GM4=,F6.4,3X,6HP3OP4=,F8.3,3X,5HPG3R=,E11.4///)
257 IF (GM4-0.95)303,262,262
262 PG4=PG3/P3OP4
265 IF(PG4-0.98*PAMB)2651,274,274
2651 LCTR=LCTR+1
2652 IF(LCTR-2)2653,2655,2660
2653 PXAA=PXL(I)
2654 PGAA=PG4
268 PXL(I)=PXL(I)-(PAMB-PG4)*C9
271 GO TO 97
2655 PXAB=PXL(I)
2656 PXL(I)=PXL(I)-( PAMB-PG4)*(PXL(I)-PXAA)/(PGAA-PG4)
PGAB=PG4
2659 GO TO 97
2660 PXAC=PXL(I)
2661 PXL(I)=PXL(I)*(PAMB-PGAB)*(PAMB-PGAA)/((PG4-PGAB)*(PG4-PGAA))
1+PXAB*(PAMB-PG4)*(PAMB-PGAA)/((PGAB-PG4)*(PGAB-PGAA))
2+PXAA*(PAMB-PG4)*(PAMB-PGAB)/((PGAA-PG4)*(PGAA-PGAB))
PXAB=PXAC
PGAB=PG4
GO TO 97
2741 PG4=PAMB
274 PG31(I)=PG3
IF(I.LT.15) GO TO 76
277 IF (I-ISTOP)278,278,311
278 IF(SIGN)76,76,279
279 IF(VL(I)-10.0)280,280,76
280 VL(I)=0.0
281 GO TO 76
303 IF(NBEST)306,306,2741
306 IF (ABSF((PG3R-PG3)/PG3)-0.02)2741,2741,3061
3061 KCTR =KCTR+1
3071 IF(KCTR-2)3074,3111,3080
3074 PXA=PXL(I)
3075 DELPA=PG3R-PG3
PXB=PXL(I)
FM4B=FM4
309 PXL(I)=PXL(I)-((PG3R-PG3)*C10)
IF(I-2)3091,3091,3092
3091 PXL(I)=(PXL(I)+4.0*PXB)/7.0
3092 PXAA=PXA
PGAA=PG3/P3OP4
310 GO TO 97
3111 PXC = PXL(I)
PXSAVE=PXL(I)
PGSAVE=PG3R-PG3
PXB=PXL(I)
DELC=PG3R-PG3

```



```

      FM4B=FM4
3073 PXL(I)=PXL(I)-(PG3R-PG3)*(PXL(I)-PXA)/(PG3R-PG3-DELPA)
      IF(I-2)3093,3093,3076
3093 PXL(I)=(PXL(I)+3.0*PXC)/4.0
3076 PXC=PXC
3077 DELB=PG3R-PG3
      PXAA=PXC
      PGAA=PG3/P3OP4
3078 GO TO 97
3080 PXC=PXL(I)
      IF(ABSF(PGSAVE)-ABSF(PG3R-PG3))3083,3083,3082
3082 PGSAVE=PG3R-PG3
      PXSAVE=PXL(I)
3083 IF((PG3R-PG3-DELC)/(PXL(I)-PXB)-100.0)3084,3084,971
3084 Z1=DELB*DELPA*(PXA-PXF)
      Z2=(PG3R-PG3)*DELPA*(PXA-PXL(I))
      Z3=(PG3R-PG3)*DELB*(PXF-PXL(I))
      FM4B=FM4
      PXB=PXL(I)
      DELC=PG3R-PG3
3081 PXL(I)=(PXL(I)*Z1-PXF*Z2+PXA*Z3)/(Z1-Z2+Z3)
      IF(PG3R-PG3)3183,3183,3180
3180 IF(DELPA)3182,3182,3181
3181 IF(PG3R-PG3-DELPA)3182,3182,3183
3182 DELPA =PG3R-PG3
      PXA=PXC
      GO TO 97
3183 DELB=PG3R-PG3
      PXF=PXC
      PXAA=PXC
      PGAA=PG3/P3OP4
3086 GO TO 97
1000 IF(NOTE)995,997,997
      995 NOTE=1
      996 GO TO 78
      997 WRITE OUTPUT TAPE 6, 999
      999 FORMAT (24H TOO MANY ITERATIONS      )
      311 GO TO 53
      312 CALL EXIT
2000 END

```

Appendix II. Definition of input variables to the computer program.

Variable Name	Definition
DELTX	Length of initial increment of liquid inflow, cm.
TON	Opening time for the inlet valve, s.
XCOLD	Length of line downstream of the inlet valve that is precooled by conduction, cm.
X4	Total length of line, cm.
DIAM	Inside diameter of pipeline, cm.
QFACT	Heat transfer enhancement factor, used in the calculation of a new temperature for a nonboiling element.
PIN	Inlet pressure, dyn/cm^2 .
PXLO	Initial assumption for the average pressure on the liquid. Used during the initial step only, dyn/cm^2 .
PAMB	Ambient pressure at the discharge of the pipeline, dyn/cm^2 .
C5	Used in the calculation of upstream gas velocity in $VG3 = VL(I) + C5 (SUMXQ/PG3)$, where $C5 = \bar{q} RT/A \lambda_{v+s}$, $\bar{q} = \pi(DIAM) (Q_B)$, Q_B is film boiling heat transfer coefficient, R is the gas constant for gaseous oxygen, A is the upstream flow area = $(\pi/4) (DIAM)^2$, and λ_{v+s} is the change in enthalpy from the liquid inlet conditions to ambient temperature gas.
C6	Used in calculation of gas, Reynolds number $REG = C6(PG3) (UG3)$, where $C6 = DIAM/RT \mu_g$, R is the gas constant, T is ambient temperature, and μ_g is the viscosity of ambient temperature gaseous oxygen.

XVALVE	Distance inlet valve is located downstream from the supply vessel, cm.
DENSL	Density of the inlet liquid oxygen, g/cm^3 .
A	Used in the calculation of the liquid and gaseous friction factor $\text{FFL} = A/\text{REL}^B$, or $\text{FFG} = A/\text{REG}^B$.
B	Also used to calculate friction factor for liquid or gas in above equations.
C1	Used to calculate liquid Reynolds number $\text{REL} = \text{C1}(\text{VL})$; where $\text{C1} = \text{DENSL} (\text{DIAM}) \mu_l$, and μ_l is the viscosity of the inflowing liquid oxygen.
C2	Used to calculate liquid friction pressure drop $\text{PFL} = \text{C2}(\text{FFL}) (\text{XL}) (\text{VL}^2)$; $\text{C2} = \text{DENSL}/2(\text{DIAM})$
C3	Used to determine heat transfer length in the two-phase liquid region; $\text{XQ} = \text{PREV}(\text{VQ}) + \text{PAMB}(1 - \text{FR}) + \text{PG3}(\text{FR}) + \text{C3}(\text{DT}) - (\text{PAMB})(\text{R})$. $\text{C3} = \text{EF} \bar{q} / A \lambda_{\text{LV}} \rho_{\text{SV}}$, where EF is an extrainment factor determining the quantity of liquid carried downstream by the vapor. A is the upstream flow area, λ_{LV} is heat of vaporization at 1 atmosphere, ρ_{SV} is the density of saturated vapor.
C8	Used to determine change in PXLO during convergence, no physical significance.
C9	Same as C8.
C10	Same as C8.

C11 Used to determine $XVAP = (XL_2 - XL_1) C11$.

$C11 = DENL/\rho_{SV}$, where ρ_{SV} is the density of saturated vapor.

C12 Used in determining flashing length,

$XFL = (XL_2 - XL_1) (TIN - TSAT) C12$.

$C12 = DENL (C_{PL}) PAMB/\lambda_{LV} \rho_{SVA}$, where C_{PL} = specific heat at constant pressure of the liquid oxygen, PAMB is the ambient pressure, λ is the latent heat of vaporization, and ρ_{SVA} is the density of saturated vapor at 1 atmosphere.

C13 Used in calculation of a new temperature for a nonboiling element.

$$C13 = \frac{0.023(\pi)(T_w - T_l)k_V}{DENS(A_i)C_{p_l}} \left(\frac{C_{p_v}\mu_V}{K_V} \right)^{0.4} \left(\frac{DIAM}{R\mu_V} \right)^{0.8}$$

where T_w is the wall temperature, k_V is the thermal conductivity of the gaseous oxygen at an average temperature, A_i is the heat transfer area $\pi(DIAM)$, C_{p_l} is the specific heat of the liquid oxygen, C_{p_v} is the specific heat of the vapor at an average temperature, μ_V , viscosity of the vapor at an average temperature, and R is the gas constant.

V SOUND Velocity of sound in ambient temperature gaseous oxygen.

IPRINT Index to start printing to help in debugging the program.

ISTOP Number of cycles program will run.

APPENDIX III. General references

The following list of references was obtained from the Cryogenic Data Center, National Bureau of Standards, Boulder, Colorado. The references were obtained by picking all entries with a keyword of "cooldown" from a computer tape containing all articles coded by the Data Center. The initial number of each article is the Cryogenic Data Center accession number and is assigned to articles as they are received. Therefore, articles with lower numbers were coded first and are usually older. The number has no significance except to the Cryogenic Data Center.

The articles were also coded as shown by the following page giving other characteristics of the retrieved articles.

APPENDIX III. General references (Cont.)

CHARACTERISTIC CODING DESIGNATIONS for CRYOGENIC LITERATURE

Categories

- A-1: Books, Reviews, Surveys, Bibliographies, Proceedings, etc.
- A-2: Properties of Solids
- A-3: Properties of Fluids
- A-4: Solid State, Theoretical, Phenomena, Basic Physics, etc.
- A-5: Cryogenic Techniques, Tricks, Unique Methods, Unusual Procedures, etc.
- A-6: Cryogenic Processes, Heat Transfer, Purification, Fluid Flow, Liquefaction, Safety Procedures, etc.
- A-7: Laboratory Equipment and Instrumentation
- A-8: Cryogenic Equipment
- A-9: General Interest Literature, News, Management, Programs, Accidents, Miscellaneous

Language

- B-1: English, B-2 French, B-3 German, B-4 Dutch, B-5 Italian, B-6 Japanese, B-7 Russian, B-8 Spanish, B-9 Other

Cryogenic Interest

- C-1: Cryogenic Temperature Range (0 to 130°K where not specifically designated in C-4 through C-7 below)
- C-2: Cryogenic Interest but not in Cryogenic Temperature Range (except where designated C-8)
- C-3: Not of Direct Cryogenic Interest
- C-4: Below 1°K
- C-5: 1 to 10°K
- C-6: 10 to 50°K
- C-7: 50 to 130°K
- C-8: 130 to 300°K

Form of Data (Omitted where not pertinent)

- D-1: Numerical Data Included
- D-2: No Data
- D-3: Graphical Data Only

Type of Article (Omitted where not pertinent)

- E-1: Experimental, Experimental and Theoretical, Original Work
- E-2: Review Article, Compilation, Correlation, Discussion
- E-3: Theoretical Only, No Specific Data Given

Availability of Document (suggested source)

- F-1: Cryogenics Division
- F-2: National Bureau of Standards
- F-3: National Technical Information Service (NTIS)
- F-4: U.S. Government Printing Office
- F-5: Defense Documentation Center (DDC) or NASA Scientific and Technical Information Facility
- F-6: Technical Libraries Generally (Published Literature)
- F-7: Technical Libraries - Special (Foreign Literature - Special Periodicals)
- F-8: Company Bulletins and Reports (Universities, Research Labs., etc.)
- F-9: Other (Patents, Theses, Translations, etc.)

Form of Document

- G-1: Published - Open Literature, Journals, etc.
- G-2: Books, Proceedings
- G-3: Company Periodicals (includes University, Foreign Gov't, State Institutions, etc.)
- G-4: Government Periodicals (U.S.)
- G-5: Company Reports, Private, Public, Gov't Contract (includes Foreign Gov't Reports)
- G-6: Government Reports (U.S.)
- G-7: University Theses, Doctoral Dissertations, Master's Theses
- G-8: Patents (U.S. and Foreign)
- G-9: Other (Unpublished, Informal, Preprints, Letters, Notes, Term Papers, Talks, etc.)

APPENDIX III. General references (Cont.)

- 7350 CRYOGENIC PIPING SYSTEM DESIGN AND INSTALLATION.
JACORS, P.R.
HEATING, PIPING, AIR CONDITIONING VOL 32, 143-156 (MAY 1960)
AF 01 01 03 E2 F6 G1 01
*COMBINED PROCESS, *HEAT TRANSFER, *FLUID FLOW, MULTI-PHASE,
SINGLE COMPONENT, GAS, LIQUID, PHASE TRANSITION, BOILING,
COOLDOWN, *SAFETY, HANDLING, TRANSFER, STORAGE,
- 9948 PRESSURIZED COOLDOWN OF CRYOGENIC TRANSFER LINES.
BURKE, J.C. BYRNES, W.P. POST, A.W. RUCCIA, F.E.
ADVANCES IN CRYOGENIC ENGINEERING VOL 4, 378-394 (PROC. 1960
CRYOGENIC ENG. CONF.) PLENUM PRESS, INC., NEW YORK (1960)
PAPER F-5, 12 FIG
AF 01 01 01 E1 F6 G2 01
*FLUID TRANSFER, PRESSURIZED TRANSFER, *COOLDOWN,
- 9855 AN INVESTIGATION OF SOME PROBLEMS CONNECTED WITH HANDLING OF
LIQUID HYDROGEN.
RICHARDS, R.J. STEWARD, W.G. JACORS, P.P. VAN GUNDY, C.A.
NATL. BUR. STANDARDS REPT. NO. 6084, (JUL 1959) 74 PP 33 FIG
1 TAB 11 REF
AF 01 01 01 F1 F2 G0 50
TRANSFER LINE, HYDROGEN, LIQUID, COOLDOWN, INSULATION, *STORAGE,
- 10334 A METHOD OF DETECTING THE FULLY COOLED STATE OF A LIQUID OXYGEN
PIPELINE.
SHUFFLEBOTHAM, N.
ROCKET PROPULSION ESTABLISHMENT, MINISTRY OF AVIATION, P.O.E.
TECH. NOTE NO. 193, 13 PP (SEP 1960) NASA N-94706, 6 FIG
NASA N62 10177
AF 01 07 01 F1 F3 G5 01
*TRANSFER LINES, *OXYGEN, SPACE APPLICATION, *COOLDOWN,
- 10637 COOL-DOWN APPARATUS FOR CRYOGENIC LIQUID CONTAINERS.
VANDER ABEEND, P.C. MANN, D.R.
U.S. PATENT NO. 2,882,694 (1959) 2 FIG
AF 01 01 03 F1 F9 G8 50
*STORAGE, *COOLDOWN,
- 11328 DESIGN OF PIPING FOR CRYOGENIC FLUIDS.
LE VALLEY, W.H. SUTTON, W.K.
ADVANCES IN CRYOGENIC ENGINEERING VOL 6, 293-321 (PROC. OF 1960
CRY. ENG. CONF.) PLENUM PRESS INC., N.Y. (1961) PAPER F-4
AF 01 06 01 F1 F6 G2 01
*FLUID TRANSFER, INSULATION, *LIQUID, *HYDROGEN, *PIPE, COOLDOWN,
- 11332 PRESSURIZED COOL-DOWN OF A CRYOGENIC LIQUID TRANSFER SYSTEM
CONTAINING VERTICAL SECTIONS.
DRAKE, E.M. RUCCIA, F.E. DUNE, J.W.
ADVANCES IN CRYOGENIC ENGINEERING VOL 6, 323-333 (PROC. OF 1960
CRY. ENG. CONF.) PLENUM PRESS INC., N.Y. (1961) PAPER F-7 6 FIG
1 TAB 7 REF
AF 01 07 01 F1 F6 G2 01
*FLUID TRANSFER, *COOLDOWN, HEAT TRANSFER COEFFICIENT, VENTING,
PRESSURIZATION,

- 11383 PROBLEMS IN COOL-DOWN OF CRYOGENIC SYSTEMS.
 ARONSON, J.C. EDESKUTY, F.J. FRETHWELL, J.H. ET AL
 ADVANCES IN CRYOGENIC ENGINEERING VOL 7, 198-206 (PROC. OF 1961
 CRY. ENG. CONF.) PLENUM PRESS INC., N.Y. (1962) PAPER F-2,
 A6 R1 C6 D1 F1 F6 G2 G3
 ROVER, *COOLDOWN, *FLUID TRANSFER, NUCLEAR REACTOR, *LIQUID,
 *HYDROGEN,
- 12985 LIQUID REQUIREMENTS FOR THE COOLDOWN OF CRYOGENIC EQUIPMENT.
 JACOBS, P.R.
 ADVANCES IN CRYOGENIC ENGINEERING VOL 8, 529-75 (PROC. 1962 CRY.
 ENG. CONF.) PLENUM PRESS, INC., N.Y. (1963) PAPER J-6
 A6 R1 C1 D3 F3 F6 G2 G3
 *COOLDOWN, *HELIUM, *HYDROGEN, *NITROGEN, *OXYGEN,
 TRANSFER LINES,
- 13315 COOLDOWN AND WARMUP OF LARGE POWDER-INSULATED DEWARs.
 KREITH, F. BROOKS, L. DEAN, J.W.
 ADVANCES IN CRYOGENIC ENGINEERING VOL 9, 536-47 (PROC. 1962 CRY.
 ENG. CONF.) PLENUM PRESS, INC., N.Y. (1963) PAPER J-7, 6 FIG
 A8 R1 C7 D3 F3 F6 G2 G3
 *COOLDOWN, DEWAR,
- 15273 HEAT INSULATOR FOR RAPIDLY COOLED LOW TEMPERATURE CHAMBERS.
 NAGAOKA, J.
 PROGRESS IN REFRIGERATION SCIENCE AND TECHNOLOGY VOL 1, 260-67
 (PROC. OF XTH INTERN. CONGR. OF REFRIG., COPENHAGEN, 1959)
 PERGAMON PRESS (1960) 9 FIG
 A8 R1 C8 D3 E1 F7 G2 G3
 *INSULATION, COOLDOWN, CORK, POLYSTYRENE, GLASS FIBER,
 SILICA GEL, STYROFOAM, FRONITE,
- 17462 TWO-PHASE FLOW DURING TRANSIENT BOILING OF HYDROGEN AND
 DETERMINATION OF NON-EQUILIBRIUM VAPOR FRACTIONS.
 CHI, J.W.H. VETERE, A.M.
 ADVANCES IN CRYOGENIC ENGINEERING VOL 9, 247-53, PROC. CRYOGENIC
 ENG. CONF., BOULDER, COLO. (AUG 1963) PAPER F-1
 A6 R1 C6 D3 F1 F8 G9 G3
 *FLUID FLOW, TWO-PHASE FLOW, *HYDROGEN, BOILING LIQUID, COOLDOWN,
- 19995 TRANSIENT TWO-PHASE HEAT TRANSFER AND FLOW CHARACTERISTICS OF
 LIQUID HYDROGEN.
 CAMPI, F.A. CHI, J.W.H. DEZURAY, F.A. HOLMGREN, J.J.
 VETERE, A.M.
 WESTINGHOUSE ELECT. CORP., ASTRONUCLEAR LAB., PITTSBURGH, PA.
 REPT. NO. WAPL-TNR-102 (APR 1963) 74 PP 25 FIG 4 TAB 9 PEF
 A6 R1 C6 D3 E1 F8 G5 G3
 TWO-PHASE FLOW, *HEAT TRANSFER, BOILING HEAT TRANSFER, *HYDROGEN,
 *GASEOUS, *LIQUID, COOLDOWN, TRANSIENT, *FLUID FLOW,
- 20259 DIGITAL SIMULATION OF A CRYOGENIC COOLDOWN EXPERIMENT.
 THURSTON, R.S.
 LOS ALAMOS SCI. LAB., N. MEX., REPT. NO. N-4-906U AND N-4-906U-A
 (JUN 1961) CONTR. NO. W-7405-ENG-36, 34 PP
 A6 R1 C6 D3 F1 F8 G5 G1
 COOLDOWN, *HYDROGEN, *LIQUID, *TRANSFER LINES, CALCULATION,
 FLUID FLOW, NUCLEAR APPLICATION,

- 20967 HEAT-TRANSFER AND FLOW DATA WITH HYDROGEN FOR NUCLEAR ROCKET SYSTEM DESIGN.
STRAIGHT, D.M. (NASA, LEWIS RES. CENTER, CLEVELAND)
PROC. ASHRAE SEMI-ANNUAL MEETING, N. Y. (FEB 11-14, 1963) 67-A4
(V. J. JOHNSON, EDITOR) AM. SOC. HEATING, REFRIG. AIR
CONDITIONING ENGRS. INC., N. Y. (1964) 12 FIG 4 PFF
MF NO. 204-E A6 P1 C6 D3 F2 F6 G2 G7
*HEAT TRANSFER, *FLUID FLOW, NUCLEAR APPLICATION,
SPACE APPLICATION, *HYDROGEN, COOLDOWN, TWO-PHASE FLOW,
- 21665 ULTRA RAPID COOLING TECHNIQUES IN THE FREEZING OF BIOLOGICAL MATERIALS.
COWLEY, C.W. TIMSON, W.J. SAWOYE, J.A. (LINDE CO.,
TONAWANDA, N. Y.)
BIODYNAMICA VOL. 8, NO. 170, 317-29 (1961) 7 FIG 2 TAB 7 PFF
A9 P1 C7 D3 F1 F6 G1 G1
*CRYOBIOLOGY, BLOOD, TISSUE, PRESERVATION, REFRIGERATION,
FREEZING, TEMPERATURE EFFECT,
A6 P1 C7 D3 F1
*HEAT TRANSFER, MULTI-PHASE, TRANSIENT, PHASE TRANSITION,
FREEZING, BOILING, NATURAL CONVECTION, NUCLEATE, FILM,
COOLDOWN
- 21672 A METHOD FOR INCREASING THE COOLING RATE IN REFRIGERATION BY IMMERSION IN LIQUID NITROGEN OR IN OTHER BOILING BATHS.
LUYET, R. (AM. FOUNDATION FOR BIOLOGICAL RES., MADISON,
WISC.)
BIODYNAMICA VOL. 8, NO. 171, 331-52 (1961) 8 FIG 5 PFF
A9 P1 C7 D3 F1 F6 G1 G1
*CRYOBIOLOGY, TISSUES, PRESERVATION, REFRIGERATION, FREEZING,
TEMPERATURE EFFECT,
A6 P1 C7 D3 F1
*HEAT TRANSFER, MULTI-PHASE, TRANSIENT, PHASE TRANSITION,
FREEZING, BOILING, NATURAL CONVECTION, NUCLEATE, FILM,
COOLDOWN
- 21902 LIQUID REQUIREMENTS FOR THE COOLDOWN OF CRYOGENIC EQUIPMENT.
JACORS, P.B.
NATL. BUR. STANDARDS CRYOGENIC ENG. LAB. NOTE NO. 61-18
(OCT 1961) 17 PP 4 FIG 1 TAB 7 PFF
A6 P1 C1 D3 F2 F1 G1 G1
*HEAT TRANSFER, MULTI-PHASE, TRANSIENT, TURBULENT, SINGLE
COMPONENT, LIQUID, GAS, PHASE TRANSITION, BOILING, COOLDOWN,
OXYGEN, NITROGEN, HYDROGEN, HELIUM
- 22104 THERMAL STRESS ANALYSIS AND COOLDOWN PROCEDURES FOR LH2 AND LO2 SYSTEMS.
EHNI, J. MCGRATH, P.J. SLEVIN, L. LOMBARDO, N.
AERJET-GENERAL CORP., AZUSA, CALIF., FINAL REPT.
153 P102 (JAN 1964) 55 PP 13 FIG 6 PFF
A6 P1 C6 D3 F1 F6 G5 G4
*HEAT TRANSFER, TRANSIENT, COOLDOWN, HYDROGEN, OXYGEN,
LIQUID

- 23345 PRESSURE OSCILLATIONS INDUCED BY FORCED HEAT TRANSFER TO TWO PHASE AND SUPERCRITICAL HYDROGEN. PRELIMINARY EXPERIMENTS.
THURSTON, R.S.
LOS ALAMOS SCI. LAB., N. MEX., REPT. NO. LAMS-3070
(1964) CONTR. NO. W-7405-ENG. 36, 93 PP 51 FIG 11 PFF
NASA N64 21055 A6 R1 C6 D3 E1 F3 G5 64
*HEAT TRANSFER, LIQUID HYDROGEN, PRESSURE SURGE, TWO-PHASE FLOW, MULTI-PHASE, COOLDOWN
- 23745 LIQUID HYDROGEN.
JACOBS, R.R.
TECHNOLOGY AND USES OF LIQUID HYDROGEN, CHAP. 4, 106-48
(SCOTT, R.R., DENTON, W.H. AND NICHOLLS, C.M. EDS.)
PERGAMON PRESS INC., NEW YORK (1964) 23 FIG 4 TAB 45 PFF
A2 R1 C6 D1 E2 F6 G2 64
*OPTICAL PROPERTY, ABSORPTION, *COPPER, *GOLD, *SILVER, *ALUMINUM, *MAGNESIUM, *CHROMIUM, *NICKEL, *RHODIUM, *LEAD, *TIN, *ZINC, *BRASS,
A2 R1 C6 D1 F2
*OPTICAL PROPERTY, ABSORPTION, *STAINLESS STEEL, *SOLDER, GLASS,
A2 R1 C6 D1 F2
*THERMAL CONDUCTIVITY, *INSULATION, PERLITE, AEROGEL, CARB-O-SIL, *MINERAL, FIBERGLASS, *FOIL, MYLAR,
A6 R1 C6 D1 F2
*HYDROGEN, *STORAGE, *STORAGE, *FLUID, TRANSFER, *TRANSPORTATION, LIQUID, COOLDOWN, PRESSURIZATION, PUMPING, TWO-PHASE FLOW
- 23903 A NITROGEN SUPPLY SYSTEM FOR POST-RUN COOLDOWN OF NUCLEAR REACTORS.
FRETWELL, J.H. (UNIV. CALIF., LOS ALAMOS SCI. LAB., LOS ALAMOS, N. MEX.)
ADVANCES IN CRYOGENIC ENGINEERING VOL 10, 402-7, PROC. CRYOGENIC ENG. CONF., PHILADELPHIA, PA. (AUG 18-21, 1964)
A6 R1 C1 D1 E1 F8 G9 64
HIGH PRESSURE, GASEOUS, COOLDOWN, NEPVA, *HEAT EXCHANGER, FIN, PERFORMANCE TEST, *NITROGEN, NUCLEAR REACTOR,
- 23907 HEAT TRANSPORT AND FLUID MOTION DURING COOL-DOWN OF SINGLE BODIES TO LOW TEMPERATURES.
FREDERICK, T.H.K. CHAPMAN, R.C. WANG, S. (CALIFORNIA UNIV., LOS ANGELES)
INTERNATIONAL ADVANCES IN CRYOGENIC ENGINEERING VOL 10, 353-360 (PROC. 1964 CRYOGENIC ENG. CONF., PT. 2, SECT. H-U) PLENUM PRESS NEW YORK (1965) PAPER T-7
A6 R1 C5 D1 E1 F8 G9 64
*HEAT TRANSFER, COOLDOWN, TRANSIENT, BOILING HEAT TRANSFER, *HELIUM, *NITROGEN, COPPER, FILM
- 23917 EFFECT OF MIST FLOW ON COOL-DOWN TEMPERATURES AND COOL-DOWN TIME.
CHI, J.W.H. (WESTINGHOUSE ELECTRIC CORP., PITTSBURGH, PA.)
ADVANCES IN CRYOGENIC ENGINEERING VOL 10, 330-340 (PROC 1964 CRYOGENIC ENG. CONF., PT. 1, SECT. A-L) PLENUM PRESS, INC., NEW YORK (1965) PAPER T-5
A6 R1 C6 D3 E1 F8 G9 64
*FLUID FLOW, *HEAT TRANSFER, BOILING HEAT TRANSFER, TWO-PHASE FLOW, TIME EFFECT, COOLDOWN, *HYDROGEN, LIQUID

- 23934 A LARGE SCALE STORAGE AND TRANSFER SYSTEM FOR DISTRIBUTION OF LIQUID HELIUM AS A REFRIGERANT MEDIUM.
GILBEAU, J.J. MARTINDALE, D.L. RUPTON, K.D. (GENERAL DYNAMICS/ASTRONAUTICS, SAN DIEGO, CALIF.)
ADVANCES IN CRYOGENIC ENGINEERING VOL 10, 428-432 (PROG 1964 CRYOGENIC ENG. CONF., PT. 1, SECT. A-L) PLENUM PRESS, INC., NEW YORK (1965) PAPER J-9
*STORAGE, COOLDOWN, PUMP, PRESSURIZATION, *HELIUM, LIQUID, SPACE APPLICATION, CENTAUR, *NITROGEN, LIQUID, TRANSFER LINES
A6 P1 C6 D3 F1 F8 G9 64
- 24701 ON THE FORMULATION AND NUMERICAL EVALUATION OF A SET OF TWO-PHASE FLOW EQUATIONS MODELLING THE COOLDOWN PROCESS.
JARVIS, STEPHEN, JR.
NATL. BUR. STANDARDS TECH. NOTE NO. 301 (JAN 1965) 49 PP
7 FIG 4 REF
*FLUID FLOW, TWO-PHASE FLOW, MODEL, MATHEMATICAL ANALYSIS, COOLDOWN,
A6 P1 C2 D3 F3 F3 G6 65
- 24832 COOL-DOWN REFRIGERATION REQUIREMENTS FOR 80 INCH CHAMBER.
RAMBERGER, J.A.
BROOKHAVEN NATL. LAB., UPTON, N. Y., REPT. NO. BNL-7496
(JAN 1960) CONTR. NO. AT(30-2)-GEN-16, 13 PP
*REFRIGERATION, COOLDOWN, BUBBLE CHAMBER, *HYDROGEN,
A6 P1 C1 D1 F1 F3 G5 60
A2 P1 C6 D1 F1
*GLASS, *THERMAL CONDUCTIVITY, SPECIFIC HEAT,
- 25240 GENERAL DESCRIPTION AND PERFORMANCE OF THE PROPLUSTON ENGINE TEST CELL (J-2A).
REEVES, J.P., JR.
ARNOLD ENG. DEVELOP. CENTER, ARNOLD AF STATION, TENN., REPT. NO. AFDC-TDR-64-138 (AUG 1964) CONTR. NO. AF 40(600)-1000, 55 PP
26 FIG 2 REF
DDC AD 444 326 MF NO. 244-F A8 P1 C1 D1 F1 F5 G5 64
*VACUUM EQUIPMENT, *TEST EQUIPMENT, SPACE APPLICATION, *HELIUM, *NITROGEN, *REFRIGERATION EQUIPMENT, CRYOPUMP, CRYOPANEL, PROPLUSTON,
A6 P1 C1 D1 F1
*COOLING PROCESS, *NITROGEN, LIQUID, *PROPLUSTON, *STORAGE, SPACE APPLICATION, *CRYOPUMPING, COOLDOWN, *HEAT TRANSFER, *HELIUM, *REFRIGERATION,
- 26310 EVALUATE ROWING AND HUMMING OF CRYOGENIC PIPELINES.
HAWORTH, W.P. BROCK, J.F. (USNP)
HEATING, PIPING, AIR CONDITIONING VOL 37, NO. 1, 179-82 (JAN 1965) 3 FIG 6 REF
*TRANSFER LINES, ROWING, MATHEMATICAL ANALYSIS, LONG DISTANCE TRANSFER, EVALUATION,
A8 P1 C1 D2 F3 F6 G1 65
*FLUID TRANSFER, TRANSFER LINES, ROWING,
A6 P1 C1 D2 F7

- 26975 AN ANALYTICAL AND EXPERIMENTAL INVESTIGATION OF THE WS 107A-2
LIQUID OXYGEN TRANSFER SYSTEM.
LITTLE, A.D. INC.
LITTLE, A. D., INC., CAMBRIDGE, MASS., TECH. REPT. NO. 4 (MAY
1960) CONTR. NO. AF 04(647)-464, 102 PP 29 FIG 8 TAB
DDC AD 454 060 A6 R1 C7 D1 E1 F5 G5 60
*TRANSFER LINES, COOLDOWN, DESIGN, TESTING, PERFORMANCE, *OXYGEN,
LIQUID, SPACE APPLICATION, GEYSERING, HEAT TRANSFER,
FLUID TRANSFER,
A6 R1 C7 D1 E1
*FLUID TRANSFER, COOLDOWN, TRANSFER LINES, TESTING, *OXYGEN,
LIQUID, *HEAT TRANSFER, *SAFETY,
- 27437 LOW TEMPERATURE STORAGE AND TRANSPORTATION OF AMMONIA.
APPLEGATE, F.L., JR. (W.R. GRACE AND CO., MEMPHIS, TENN.)
AIChE 57TH ANN. MEETING, BOSTON, MASS. (DEC 6-10, 1964)
PAPER NO. 14D, 13 PP
A6 R1 C8 D3 E1 F6 G1 64
*STORAGE, *TRANSPORTATION, AMMONIA, LIQUID, COOLDOWN, SAFETY,
OVERWATER, ECONOMIC FACTOR,
- 29268 PREDICTING PRESSURE SURGES IN CRYOGENIC TRANSFER LINES DURING
COOL DOWN.
MODERN REFRIGERATION
MOD. REFRIG. VOL. 68, NO. 1, 460-1 (MAY 1965)
A6 R1 C7 D1 E2 F7 G1 65
*FLUID TRANSFER, COOLDOWN, TRANSFER LINES, PRESSURE SURGE,
REVIEW, *NITROGEN, LIQUID,
- 30527 SORPTION PUMP APPARATUS.
NEEDHAM, C.G. WOOD, H.K. (VARIAN ASSOCIATES, PALO ALTO, CALIF.)
U. S. PATENT 3,172,745 (MAR 9, 1965) 2 PP 3 FIG 4 REF
A6 R1 C2 D2 E1 F9 G8 65
*VACUUM EQUIPMENT, ABSORPTION PUMP, PRECOOLING, COOLDOWN, PATENT,
- 31041 SLUG FLOW AND FILM BOILING OF HYDROGEN.
CHI, J.W.H. (WESTINGHOUSE ELECTRIC CORP., ASTRONUCLEAR LAB.,
PITTSBURGH, PA.)
ASME WINTER ANNUAL MEETING, CHICAGO (NOV 7-11, 1965) PAPER NO.
65-WA/HT-32, 12 PP 7 REF
A6 R1 C6 D1 E1 F8 G9 65
*HYDROGEN, *FLUID FLOW, FILM BOILING, SLUG FLOW, *LIQUID,
*HEAT TRANSFER, COOLDOWN,
- 31775 LIQUID HELIUM GSE FOR CENTAUR TURBOPUMP CHILDDOWN.
GILBEAU, J.J. MARTINDALE, D.L. BURTON, K.R. (GEN DYNAMICS/
CONVAIR, SAN DIEGO, CALIF.)
AIAA ANNUAL MEETING, 2ND, SAN FRANCISCO (JUL 26-29, 1965)
PAPER NO. 65-523, 12 PP
A6 R1 C5 D1 E1 F8 G9 65
*STORAGE, *HANDLING, *LIQUID HELIUM, CENTAUR, *PUMPS, COOLDOWN,
GROUND SUPPORT EQUIPMENT, SPACE APPLICATION,

- 31814 COOLDOWN OF INSULATED METAL TUBES TO CRYOGENIC TEMPERATURES.
MADDOX, J.P. FREDERKING, T.H.K. (CALIF. UNIV., LOS ANGELES)
ADVANCES IN CRYOGENIC ENGINEERING VOL. 11, 536-46 (PROC.
1965 CRYOGENIC ENG. CONF.) PLENUM PRESS, INC.,
NEW YORK (1966) PAPER H-5
A8 R1 C1 D3 F1 F8 G9 65
*HEAT TRANSFER, *INSULATION, PIPE, COOLDOWN, METAL, COEFFICIENT,
*LIQUID NITROGEN, COATED, FILM, CYLINDER,
- 31817 CHILLOWDOWN AND STORAGE LOSSES OF LARGE LIQUID HYDROGEN STORAGE
DEWARS.
LIEBENBERG, D.H. STOKES, R.W. EDESKUTY, F.J. (LOS ALAMOS SCI.
LAB., NEW MEX.)
ADVANCES IN CRYOGENIC ENGINEERING VOL. 11, 554-60 (PROC.
1965 CRYOGENIC ENG. CONF.) PLENUM PRESS, INC.,
NEW YORK (1966) PAPER H-7
A8 R1 C6 D1 F1 F8 G9 65
*STORAGE, *DEWAR, LOSSES, *LIQUID HYDROGEN, KIWI, COOLDOWN,
COMPARISON, MATHEMATICAL ANALYSIS, BOILOFF,
- 32071 PRESSURE SURGE IN CRYOGENIC LINES.
NATIONAL BUREAU OF STANDARDS TECHNICAL NEWS BULLETIN
NATL. BUR. STD. TECH. NEWS BULL. VOL 49, NO. 8, 129 (AUG 1965)
A8 R1 C7 D3 F1 F4 G1 65
*FLUID FLOW, *PRESSURE, SURGE, COOLDOWN, CRYOGENIC APPLICATION,
*TRANSFER LINE, PRESSURE SURGE,
- 33529 ANALYSIS OF COMPLEX CRYOGENIC CYCLES.
BOLSTAD, M.M. (MISSOURI UNIV., COLUMBIA, DEPT. OF MECHANICAL
ENGINEERING)
ASHRAE JOURNAL VOL 7, NO. 12, 29-32 (DEC 1965) 6 FIG
A6 R1 C1 D3 F3 F6 G1 65
*CYCLE, CLAUDE CYCLE, COOLDOWN, *LIQUEFACTION,
- 34315 LOW TEMPERATURE AND CRYOGENIC STEELS.
UNITED STATES STEEL CORPORATION
UNITED STATES STEEL CORP., PITTSBURGH, PA., MATERIALS MANUAL
ADUSS 01-1206 (FEB 1966) 194 PP
A2 R1 C7 D1 F2 F8 G5 66
*STEEL, NINE PERCENT NICKEL, *THERMAL EXPANSION, STAINLESS 304,
*THERMAL CONDUCTIVITY, *STAINLESS STEEL, *SPECIFIC HEAT, WELD,
*TENSILE PROPERTY, IMPACT TEST, *NOTCHED SPECIMEN, NICKEL STEEL,
A2 R1 C7 D1 E2
*TENSILE PROPERTY, *STAINLESS STEEL, STAINLESS 301,
STAINLESS 302, STAINLESS 310, STAINLESS 303, STAINLESS 316,
STAINLESS 347, STAINLESS 321,
A2 R1 C1 D1 E2
*THERMAL EXPANSION, STAINLESS 301, STAINLESS 302, *ALUMINUM,
*COPPER, NINE PERCENT NICKEL,
A2 R1 C7 D1 F2
*INSULATION, *THERMAL CONDUCTIVITY, PERLITE, AEROGEL,
GLASS FIBER, FOAM, POLYSTYRENE, POLYURETHANE, SUPERINSULATION,
A8 R1 C7 D1 F2
STEEL, STAINLESS STEEL, FABRICATION, COST, WELDS, AVAILABILITY,
COOLDOWN, *STOPAGE,
A2 R1 C8 D1 E1
NICKEL STEEL, CARBON STEEL, MARTENSITIC TRANSFORMATION,
GRAIN SIZE, *PHASE TRANSITION PROPERTY,

- 35469 FLOW SYSTEM STARTUP OF A FULL-SCALE SIMULATED NUCLEAR ROCKET ENGINE.
COLMERY, R.H. POWERS, A.G. (LEWIS RESEARCH CENTER, NATIONAL AERONAUTICS AND SPACE ADMINISTRATION, CLEVELAND, OHIO)
IEEE TRANS. NUCL. SCI. VOL NS-13, NO. 1, 658-62 (FEB 1966)
A8 R1 C6 D F F6 G1 66
*FLUID FLOW, SIMULATION, NUCLEAR APPLICATION, ROCKET, ENGINE, FLOW, COOLDOWN, SYSTEM, OSCILLATORY EFFECT, TEST RESULTS, SPACE APPLICATION,
- 36242 RECOMMENDED COOLDOWN PROCEDURES FOR F-70NF CRYOGENIC PIPING SYSTEMS.
AEROJET-GENERAL CORPORATION
AEROJET-GENERAL CORP., COVINA, CALIF., AETRON DIV., REPT. NO. NASA-CR-70517 (DEC 1963) CONTR. NO. NAS8-4014 AND NAS8-4015, 21 PP
NASA N66 18162 A6 R1 C6 D3 E2 F3 G5 63
*COOLING PROCESS, *TRANSFER LINE, COOLDOWN, PROPULSION, SPACE APPLICATION, TEST FACILITY, HYDROGEN, OXYGEN, LIQUID,
- 36573 COOL-DOWN TIME OF INSTALLATION INCORPORATING STIRLING CYCLE REFRIGERATOR.
DAMSZ, G.J. (NORTH AMERICAN PHILIPS CO., INC., NEW YORK, N. Y.)
U. S. PATENT 3,220,200 (NOV 1965) 2 PP 3 FIG 4 REF
A8 R1 C1 D3 F1 F9 G8 65
*REFRIGERATION, REFRIGERATOR, STIRLING, COOLDOWN, PATENT,
- 36590 PRECOOLING AIR IN OXYGEN PRODUCTION
KEITH, P.C. (HYDROCARBON RESEARCH, INC., NEW YORK, N. Y.)
U. S. PATENT NO. 2,643,527 (JUN 1953) 6 PP 3 FIG 7 REF
A8 R1 C7 D3 E1 F9 G8 57
*REFRIGERATION, *GAS LIQUEFACTION, *GAS SEPARATION, LIQUEFIED GAS RECTIFICATION, AIR, PATENT, COOLDOWN, PRECOOLING, *HEAT EXCHANGER, REVERSING, *GAS PURIFICATION, REMOVAL, WATER VAPOR, CARBON DIOXIDE,
- 37130 COMMERCIAL HANDLING OF LIQUID HELIUM DEWARs.
VAN MEERBEKE, R.C. (AIR REDUCTION CO. INC., JERSEY CITY, N. J.)
ADVANCES IN CRYOGENIC ENGINEERING VOL 12, 646-56 (PROC. OF CRYOGENIC ENG. CONF.) PLENUM PRESS, INC., NEW YORK (1967)
PAPER J-2 AND BULL. IIR ANNEX 1966-5, 303-17 (MEETING OF COMM. 1, BOULDER, COLO., JUN 15-17, 1966)
A8 R1 C5 D3 F2 F8 G9 66
*DEWAR, HANDLING, TRANSPORTATION, LIQUID, HELIUM, *SAFETY, HAZARDS, VALVE, RELIEF VALVE, *STORAGE, LOSSES, TRANSFER, COOLDOWN, HEAT LEAK,
- 37156 COOLDOWN OF SHROUDED SPHERICAL VESSELS IN LIQUID NITROGEN.
MANSON, L. (ROCKETDYNE, CANOGA PARK, CALIF.)
CRYOGENIC ENGINEERING CONF. 12TH, BOULDER, COLO. (JUN 13-15, 1966) PAPER C-1, 20 PP
A8 R1 C7 D E F8 G9 66
*REFRIGERATION, *HEAT TRANSFER, *STORAGE EQUIPMENT, CONTAINER, COOLDOWN, SPHERE, HEAT TRANSFER COEFFICIENT, GEOMETRY EFFECT, SURFACE EFFECT, TEFLON, BOILING HEAT TRANSFER, NITROGEN, LIQUID,

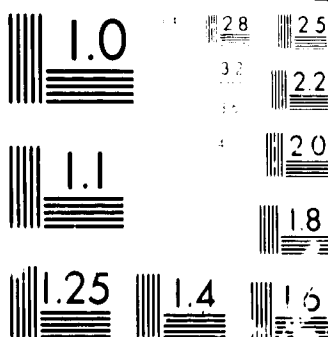
- 37902 THERMAL CONTROL OF LIQUID H₂/O₂ ROCKET ENGINES AND VEHICLE IMPACT.
WINSTEAD, T.W. (MARSHALL SPACE FLIGHT CENTER, NATIONAL AERONAUTICS AND SPACE ADMINISTRATION, HUNTSVILLE, ALA.)
AIAA PROPULSION JOINT SPECIALIST CONF. 2ND, COLORADO SPRINGS, COLO. (JUN 13-17, 1966) PAPER NO. 66-562
AB R1 C6 D1 E1 F8 G9 66
*PROPULSION, CONTROL, ROCKET, ENGINE, PROPELLANT, *PUMPS, HYDROGEN, OXYGEN, LIQUID, SPACE APPLICATION, COOLING, NPSH, COOLDOWN,
- 37907 A COMPARISON OF COOLDOWN TIME BETWEEN INTERNALLY COATED AND UNCOATED PROPELLANT LINES.
LEONHARD, K.E. GETTY, R.C. FRANKS, D.F. (GENERAL DYNAMICS/CONVAIR, SAN DIEGO, CALIF.)
ADVANCES IN CRYOGENIC ENGINEERING VOL 12, 731-9 (PROC. 1966 CRYOGENIC ENG. CONF.) PLENUM PRESS, INC., NEW YORK (1967) PAPER NO. F-2
AF R1 C D E F8 G9 66
*TRANSFER LINES, INSULATED, COATING, SURFACE, UNINSULATED, TESTING, COOLDOWN, COMPARISON, APPARATUS, STAINLESS STEEL, TEST RESULTS, TIME CONSTANT,
- 38620 OPTIMIZATION OF COOL-DOWN OF SOLIDS IN LOW BOILING POINT LIQUIDS.
FREDERICK, T.H.K. CHAPMAN, P.C. (CALIFORNIA UNIV., LOS ANGELES) RULL. IIR ANNEXE 1965-2, PP 277-84 (MEETING OF COMMISSION 1, GRENABLE, FRANCE, JUN 8-11, 1965)
A6 R1 C5 D1 E1 F7 G2 65
*HEAT TRANSFER, COOLDOWN, SOLID, HEAT LOSS, RATE, COATING, OPTIMIZATION, THICKNESS, CRYOGENICALLY COOLED, EFFECT, MATHEMATICAL ANALYSIS,
- 39273 COOLDOWN OF CRYOGENIC TRANSFER LINES.
LIEBENBERG, D.H.
LOS ALAMOS SCIENTIFIC LAB., N. MEX., REPT. NO. LA-3426 (NOV 1965) CONTR. NO. W-7405-ENG-36, 22 PP
NSA 66 1366A
AB R1 C6 D E F3 G5 65
*TRANSFER LINES, HYDROGEN, LIQUID, COOLDOWN,
- 39289 COOLDOWN OF LARGE-DIAMETER LIQUID HYDROGEN AND LIQUID OXYGEN LINES.
COMMANDER, J.C. SCHWARTZ, M.H.
AEROJET-GENERAL CORP., SACRAMENTO, CALIF., REPT. NO. NASA-CR-54809, AGO-8800-54 (APR 1966) CONTR. NO. NAS3-2555, 50 PP
NASA N66 25246 ME NO. NASA
AF R1 C6 D1 E1 F3 G5 66
*TRANSFER LINE, COOLDOWN, HIGH CAPACITY, HYDROGEN, OXYGEN, LIQUID, *TEST EQUIPMENT, TESTING, FACILITY, MATHEMATICAL ANALYSIS, THEORY,
- 39575 DENSE GAS HELIUM REFRIGERATOR.
HOOD, C.R., JR. (CRYOVAC, INC., COLUMBUS, OHIO)
U.S. PATENT 3,269,137 (AUG 1966).
AB R1 C5 D3 E1 F9 G8 66
*REFRIGERATION, HELIUM REFRIGERATOR, HIGH SPEED, COOLDOWN, HIGH MAGNITUDE, EFFICIENCY, PATENT,

- 40826 COOLDOWN OF CRYOGENIC TRANSFER LINES - AN EXPERIMENTAL REPORT.
BRENNAN, J.A. BRENTARI, E.P. SMITH, R.V. STEWARD, W.G.
NATIONAL BUREAU OF STANDARDS, BOULDER, COLO., REPT. NO. 9264
(NOV 1966) 69 PP 51 FIG 1 TAB 3 REF
A6 R1 C6 D F F2 G9 66
*TRANSFER LINE, COOLDOWN, TESTING, LIQUID NITROGEN,
LIQUID HYDROGEN, *HEAT TRANSFER, PRESSURE EFFECT, HEAT TRANSFER,
TEMPERATURE EFFECT,
- 40851 SUMMARY REVIEW OF SEMINAR S-12.
COMMANDER, J.C. (AEROJET-GENERAL CORP.)
CRYOGENIC ENG. NEWS VOL 1, NO. 14, 40 (DEC 1966)
A6 R1 C6 D2 E2 F6 G1 66
*TRANSFER LINE, COOLDOWN, TECHNIQUE, COOLING,
- 40885 A METHOD OF INCREASING HEAT TRANSFER TO SPACE CHAMBER CRYOPANELS.
ALLEN, L.D. (MANNED SPACECRAFT CENTER, NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION, HOUSTON, TEX.)
ADVANCES IN CRYOGENIC ENGINEERING VOL 11, 547-53 (PROC. 1965
CRYOGENIC ENG. CONF.) PLENUM PRESS, INC., NEW YORK (1966)
PAPER H-6
A6 R1 C7 D1 F1 F6 G2 65
*HEAT TRANSFER, CRYOPANEL, SPACE SIMULATOR, SURFACE COATING,
TEFLON, COOLDOWN,
- 41594 A STUDY ON CLOSED-LOOP CRYOGENIC SYSTEMS FOR TELEMETRY MASER
AMPLIFIERS.
LASHMET, P.K. MEIER, P.N. SIEGPIST, G.W.
AIR PRODUCTS AND CHEMICALS, INC., ALLENTOWN, PA., REPT. NO.
ASD-TR-61-311 (AUG 1961) CONTR. NO. AF 33(616)-7231 98 PP
DDC AD 800 405
A6 R1 C5 D1 F1 F5 G5 61
*REFRIGERATION, *CYCLE, *HELIUM, CLOSED CYCLE, MASER, *HYDROGEN,
JOULE-THOMSON COOLING, MINITATURE DEVICE, CONTAMINATION, COOLDOWN,
STIRLING CYCLE, COMPRESSOR, EXPANSTION ENGINE, *NITROGEN,
A2 R1 C7 D1 F2
*INSULATION, *THERMAL CONDUCTIVITY, PERLITE, SANTOCFL, FOAM,
FIBERGLASS, POLYURETHANE, POLYSTYRENE, SUPERINSULATION,
PRESSURE EFFECT, VACUUM,
A2 R1 C7 D1 F2
*EMISSIVITY, SURFACE TREATMENT, *ALUMINUM, *OXIDE, ALUMINUM,
*BRASS, *CHROMIUM, *COPPER, COPPER, *GOLD, *GLASS, *NICKEL,
*SILVER, STAINLESS 302,
- 42356 SUMMARY OF OBSERVED RESULTS WHEN CHILLING THE M-1 FUEL TURBOPUMP
TO LIQUID HYDROGEN TEMPERATURE.
PITZER, J.A.
AEROJET-GENERAL CORP., SACRAMENTO, CALIF., LIQUID ROCKET OPERA-
TIONS, REPT. NO. AGC-8800-65, NASA-CR-54828 (JUN 1966) CONTR.
NO. NAS3-2555 80 PP
NASA N66-25585
A6 R1 C6 D F F3 G5 66
*HEAT TRANSFER, *REFRIGERATION, *PUMP, COOLING, COOLDOWN,
CRYOGENICALLY COOLED, PROPELLANT, ROCKET FUEL,
TEMPERATURE EFFECT, OPERATION, TESTING,

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- 43454 ARGON PURGE GAS COOLED BY CHILL BOX.
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION, WASHINGTON, D.C.,
TECH. REPT. 866-10153 (APP 1966)
NASA 866-10153 AR R1 CR D3 F1 F7 G6 66
*REFRIGERATION, ICE, COLD BOX, COOLDOWN, *HEAT EXCHANGER, COIL,
ARGON, GAS, PURGING,
- 43697 METHOD FOR COOLING AND FILLING LIQUEFIED GAS TRANSPORT AND
STORAGE TANKS.
NONNECKE, F.A. GOTTSCHE, E.H.W. (MCMULLEN (JOHN J.) ASSOCIATES,
INC., NEW YORK)
U.S. PATENT 3,313,116 (APP 1967)
AR R1 C1 D3 F1 F9 G8 67
*TRANSPORTATION, *STORAGE, SHIP, FILLING, COOLDOWN, METHOD,
PROCESS, PATENT, HANDLING, LNG, LIQUEFIED GASES,
- 43946 METHOD OF THERMAL DESIGN OF EVAPORATOR FOR COOLING NATURAL GAS.
RUDAGYAN, F.A.
GAZ. PROM. VOL 11, NO. 11, 7-10 (NOV 1966)
MF NO. 704-L AR R7 C7 D1 F1 F7 G1 66
*SEPARATION, NATURAL GAS, DISTILLATION EQUIPMENT, *REFRIGERATION,
EVAPORATIVE COOLER, COOLDOWN, DESIGN, MATHEMATICAL ANALYSIS,
- 44280 TWO-PHASE SPRAY SYSTEM FOR FILLING TANKS.
CROWNOVER, C.F. (CONCH INTERNATIONAL METHANE LTD., NASSAU,
BAHAMAS)
U. S. PATENT 3,311,305 (MAR 1967)
AR R1 C1 D3 F1 F9 G8 67
*TRANSFER LINE, *STORAGE, TANK, FILLING, COOLDOWN, PATENT,
- 45963 LOW TEMPERATURE REFRIGERATION IN ETHYLENE PLANTS.
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*LOX, CLEANING,
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A6 R1 C5 D1 F2
COOLDOWN, *TRANSFER LINE, STAINLESS STEEL, *INSULATING SUPPORT, *HEAT TRANSFER, ELECTRIC LEADS, RADIATION SHIELD, *COOLING PROCESS,
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A8 R1 C5 D1 F1
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 A6 R1 C6 D1 E3
 *INSULATION, *HYDROGEN, LIQUID, SLUSH, COMPARISON, OPTIMIZATION,
 SPACE APPLICATION, SATURN, ORBITAL TANKER, *STORAGE, VENTING,
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 A7 R1 C6 D2 E2
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*LIQUID HYDROGEN, DETONATION, PROTECTION, *DEWAR, LEAK, PURGING,
COMBUSTION, CYLINDER,
A6 R1 C6 D1 E2
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CHECK VALVE,
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WATER VAPOR, THICKNESS, CRYSTAL STRUCTURE, TIME EFFECT, HUMIDITY,
COOLDOWN,
A7 R1 C7 D1 E1
*HEAT TRANSFER, FROST FORMATION, *MEASUREMENT, THICKNESS,
THERMAL CONDUCTIVITY,
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DESIGN, *HEAT TRANSFER, NUCLEATE BOILING, POOL BOILING, CHOKING,
FILM BOILING, QUALITY, VELOCITY, *INSULATION,
A6 R1 C1 D3 E3
*HEAT TRANSFER, *GASEOUS, *HELIUM, *HYDROGEN,
A3 R1 C6 D1 E1
COOLDOWN, TIME EFFECT, *THERMAL PROPERTY, HEAT TRANSFER, MONEL,
STAINLESS 321, ALUMINUM ALLOY, TITANIUM ALLOY, *COPPER, TUBING,
A6 R1 C6 D1 E1
*LOX, *PUMP, TURBINE, SURFACE PROPERTY, *LIQUID HYDROGEN,
A3 R1 C6 D1 F1
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VAPOR-LIQUID EQUILIBRIUM, SATURATION CURVE,
A2 R1 C6 D1 F1
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A2 R1 C1 D1 E2
*INSULATION, *THERMAL CONDUCTIVITY, PERLITE, FIBERGLASS, FOAM,
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*LIQUID NITROGEN, *SEAL,
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